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Microwave Beam Shaping for Processing Geometrically Complex Ceramics

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14. ABSTRACT

Selective volumetric material manufacturing, a type of additive manufacturing, is the creation of a solid object or surface within a volume of loose material, such as precursor powder. Interacting with the loose material is done on the length of the intended object, unlike typical additive processing techniques, which build the object path-by-path and layer-by-layer. This means that the intended volumetric object can be created with one or several discrete events as opposed to building the object using multiple mm or um layers. One example of this volumetric processing is the use of a high energy microwave to generate heat in a specified shape within a volume of loose dielectric ceramic powder. The microwave beam deposits energy into desired locations within the volume, sintering these locations into an object or surface of desired shape. Knowledge of the electromagnetic, thermal, and mechanical properties of the microwave system and precursor materials used are essential for predictively generating the desired shape and make volumetric material manufacturing non-trivial. This work is a direct extension of our previous work and represents our ongoing efforts toward manufacturing volumetric parts utilizing microwave sintering of ceramics.

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EXECUTIVE SUMMARY

The purpose of this report is to describe the progress of the NRL 6.1 Core program titled “Microwave Beam Shaping for Processing Geometrically Complex Ceramics”. This program attempts to lay the scientific foundations of shaping microwave beams for selectively heating ceramic powders to the sintering temperature and thus enabling forming complex geometric shapes after the volume of the ceramic has reached room temperature. This work has emphasized in the computational infrastructure used to predict beam shaping, both in free space and withing the volume of the ceramic powder, and also in initial experimental validation through the magnetron irradiation of ceramics.

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MICROWAVE BEAM SHAPING FOR PROCESSING GEOMETRICALLY COMPLEX CERAMICS

1. ACHIEVED PROGRESS

1.1 Summary of the technical progress achieved

To achieve the objectives of this program we have developed a multiphysics framework for modeling microwave energy transfer through heterogeneous media, and also experimental procedures for irradiating ceramic powders using magnetrons. The proposed method utilizes microwave radiation to penetrate and heat the material at desired locations anywhere within its domain. This concept offers the possibility of a new family of additive manufacturing methods for high-performance materials. The simulations produced by exercising this framework indicate that employing a grating pattern with regions of discretely varying conductivity and permittivity elements placed between the microwave source and the volume of the ceramic powder to be processed can lead to the capability of controlling the shape of volumetric heat localization. The experimental process was first established on an 85GHz gyrotron, through the development of process control software, and then repurposed for a 2.45GHz magnetron system.

1.2 Technical progress for each of the technical areas

The efforts of this project have focused on several technical areas. These are development of beam shaping for materials processing through holographic and inverse approaches, control process development, material properties identification and characterization, and fabrication.

A multiphysics approach was essential to set the foundation of selective volumetric sintering on two topics: microwave sintering and microwave propagation control. Microwave sintering requires a multiphysics approach because the material properties of the precursor sample powders are complex and nonlinear. As the microwave deposits energy into the powder, the properties change, thereby affecting the microwave propagation. This creates a nonlinear feedback loop where the ceramic density, electric permittivity, loss tangent, specific heat capacity, and thermal conductivity all change as a function of temperature. Therefore this problem must be modeled using a multiphysics approach by coupling thermal physics, electric fields, microwave propagation, and the equation of motion in its stress form. Similarly, microwave propagation control requires a multiphysics approach to couple the incident microwave to the propagation modification technique. For example: using a plasma cell for modulating the incident plasma beam requires coupling an electrically driven plasma system to the microwave propagation. In this coupled system, the microwave and electrical driving system excite the plasma, and the excited plasma affects the microwave propagation. To address the need of predicting the shaper of the microwave beam we implemented two approaches: one based on holographic principles and another based on linerization of the coupled thermal/irradiation physics.

This project utilized two unique experimental systems for microwave production. Initial work utilized a 85GHz Gyrotron to irradiate compressed pellets of Zirconia in free space. However, in year 3 of the project,

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the Gyrotron tube was damaged and the system could not be recovered. Therefore, work was transitioned to a 2.45GHz magnetron irradiating loose Barium Titanate (BTO) within a TE_{106} resonant cavity waveguide setup. Both 85 and 2.45GHz systems were modeled using a multiphysics framework based on finite element analysis (FEA). This was done in order to clearly understand the microwave propagation within the respective systems as well as the physical mechanisms for heat transfer from microwave to ceramic precursor powder. As the precursor powders were heated they underwent sintering, a process where atoms migrate into gaps thereby solidifying the previously porous material.

1.2.1 Development of Beam Shaping for Materials Processing

Below are the major developments under the development of beam shaping for materials processing technical area performed by the members of Code 6394.

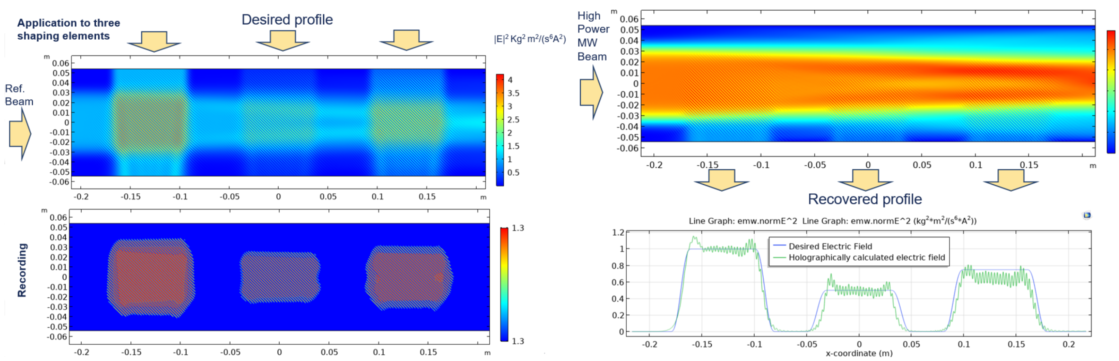
Theoretical Coupled Multiphysics Framework

The coupled electromagnetic, thermal PDEs were properly considered in a multiphysics finite element model. The coupled character of these physics is essential due to the temperature dependent dielectric properties of the BTO sample powder. Specifically, the powder density, specific heat, dielectric constant and loss tangent are nonlinear with respect to temperature. So as the microwave energy propagates through the dielectric powder, it deposits heat via Joule heating, which changes the dielectric constants, which then changes how the energy propagates. This cycle is dynamic and nonlinear, requiring the coupling of electromagnetic, thermal, and Joule heating PDEs, along with the sample properties. A similar coupling of PDEs was done for the plasma cell to incident microwave system.

Inverse Beam Shaper Identification Theory

The shaping of the microwave beam via its interaction with the grating is possible because of the proper spatial arrangement involving alternating presence of conductive and dielectric elements. This arrangement can be dynamically controlled using mechanical devices such as electromechanically controlled positioning devises. A more flexible, economical and effective option though is the utilization of an array comprised by individually addressable plasma elements. The plasma array can be controlled by the pressure, voltage, species, excitation, and geometry of each plasma element, thereby enabling the control of the spatial distribution of the dielectric and conductive properties that in turn can enable the desired microwave beam shape. This beam shaping can be either amplitude or phase based. The latter is particularly attractive, because beam energy loss can be minimized.

While sintering an arbitrary shape in the powder volume is straightforward, sintering a desired shape remains challenging. To address this, the incident microwave energy must be perturbed so that heat is localized at predetermined locations. We propose the use of a conductive mask placed in the microwave cavity to cause energy signal interference within the powder volume. In order to determine the mask pattern required to make a sintered shape, virtual experiments must be conducted. To compute the arrangement of the pattern, we developed two approaches: (a) A 3D and 2D FEA were created and multiple arbitrary masks are used to generate heat patterns in the powder volume in a forward problem; this data is then used to solve the inverse problem; given a shape, determine the mask pattern required to generate that shape in the powder volume, and (b) A computational holography approach was developed to generate grating patterns by virtue of recording of a desired beam profile on resulting from its intersection with the Gaussian output beam of a microwave generating device. An example computational holography (recording and irradiation steps) are shown in Fig. 1.



(a) Computational holography recording step. (b) Computational holography irradiation step.

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Fig. 1—Computational holography for shaping microwave beams for the purpose of ceramic powder sintering

1.2.2 Control Process Development

Below are the major developments of the control process design and implementation technical area performed by the members of Code 6394 and 6790.

Incident Microwave Characterization and Profile Identification

The NRL 85GHz gyrotron was used to conduct these experiments. The gyrotron power output was monitored during experiments by an actively coupled antenna and spectrometer to confirm frequency tolerance within 10MHz. Power output was calibrated using a water cooled beam dump where the input and output temperatures were monitored in addition to the flow rate. This calibration was conducted by diverting the microwave path to the beam dump prior to the experimental setup involving the sample powder. After calibration, the microwave power was focused to approximately 3cm diameter profile using a chilled boron nitride lens. The 3.5mm wavelength was measured using a burn paper screen at powers less than 100W, producing a vertical interference pattern as expected from the gyrotron construction. This information allowed accurate modeling of the system in 2D and 3D using FEA.

For the purpose of transitioning the approach to a 2.45 GHz magnetron system, it was required to confine microwave power to a waveguide resonant cavity. A resonant cavity was created within the waveguide by utilizing a 3-stub tuner used for impedance matching and defining the front of the microwave cavity. Inside the cavity, a bi-directional power coupler was used to measure power propagation in both directions through the waveguide. The sintering target was placed inside a 21.6 cm long WR-340 waveguide (4.31 x 8.63 cm cross section) with low and high frequency cutoffs at 1.736 GHz and 3.471 GHz respectively. The WR-340 waveguide terminates at a variable short, creating the back of the microwave cavity. This short is a flat metal wall acting as a perfect conductor to reflect the incident microwave radiation. The short could be extended up to 10.2 cm. The incident power created a standing wave within the resonant cavity with powers 2-3 orders of magnitude higher than the incident power.

The 2.45GHz magneton output was directly measured by waveguide coupler using a spectrum analyzer to determine both frequency and power output. During experiments, microwave power directional power couplers were utilized to measure the forward and backward power components at different parts of the waveguide including within the resonant cavity. The standing wave was measured experimentally by moving an empty boron nitride crucible along the waveguide section. When the crucible was at the peak electric field, power was attenuated as the crucible was heated, but when the crucible was at peak magnetic field, power was not attenuated. The expected sine curve matched the calculated waveguide wavelength of 17.2cm.

Additionally, the microwave cavity was optimized by modeling the system. The free parameters in the cavity are the position of the variable short and the insertion distance of the 3 stubs in the 3-stub tuner. Experimentally adjusting these parameters can create localized power maximums that correspond to harmonics, but the global maximum is time consuming to find using experimental techniques. This is because local optimums span several different 10-100 μm positions within parameter spaces of 3.4 cm (stubs) and 10.2 cm (short). A virtual test was done in hours instead of days and proved to be a good technique for finding the global maximum power containment of the microwave cavity.

Temperature ΔT Control

Conventional sintering is typically done in a furnace where the temperature is slowly increased or decreased. This temperature over time gradient is referred to as ΔT and is the subject of much research for each ceramic powder. The heating rate in microwave sintering can be much higher than a furnace, therefore it is important to monitor and control the heating rate. In order to control ΔT, the temperature sensor outputs had to form a feedback control loop with the microwave power output. For both the 85GHz gyrotron and 2.45GHz magnetron, pyrometers were fed into a computerized control system that we developed, so that predetermined temperature profiles could be examined.

Gyrotron Control Upgrades

Improvements were made to the electronics and control systems of the 85GHz gyrotron. At the start of the project, startup protocols, safety interlocks, power control, and pyrometer data were all operating on separate computer programs. These programs were combined into a master program. Additionally, the master program allowed multi-step programming of the gyrotron power output or sample temperature. This meant that strict ΔT profiles could be followed while sintering, including ”resting” steps or very gradual increases or decreases. In this way, the system was automated and sintering processes of several hours could be done. Best sintering results were found for yittria stabilized zirconia (YSZ) when positive ΔT was less than 30K/min with rest steps of 10min at approximately 1700K and 2100K and then cooling at 10K/min to room temperature.

Beam Shaping with DC Plasma

The relationship between microwave signals and plasma has been widely studied across the literature. The free-space, non-semiconductor related studies generally focus on several key areas: microwave sustained plasma, chemical vapor deposition, plasma creation with microwaves, or generation of microwave from plasma. Generally, a microwave is used to heat a plasma to sustain a desired reaction or create a desired optic or thermal profile. Conversely, an AC-coupled plasma can be used to generate, modify, or amplify microwaves.

Instead of this classical approach, we proposed to modulate the incident microwave beam using plasma. The goal is to pass a microwave signal through a plasma element, which is externally adjusted to change the propagating direction, phase, or intensity of the microwave. An array of these plasma elements can then be used to shape the microwave beam, focusing it, and creating unique beam geometries. The modified beam can then be used for material processing, for example, the sintering of ceramics due to the induced Joule heating on a material of interest. During this process, the microwave locally deposits energy into a ceramic powder, thereby heating it and consequently inducing sintering into a specific shape.

Coupling the electromagnetic propagation to the plasma physics in a multiphysics framework allowed the modeling of a conventional argon mercury plasma, such as present in a commercial flash lamp. Inserting the plasma cell into the 2.45GHz waveguide was modeled the physics 2D and 3D frameworks and confirmed that a plasma element can be used to modulate microwave propagation. Specifically, 600V DC supplied to the plasma cell electrodes resulted in plasma electron densities over $1^{20}m^{-3}$, capable of reflecting nearly all of the incident microwave power. This reflection is limited to the waveguide profile occupied by the plasma cell footprint. An array of these plasma cells can then be used for microwave beam shaping in order to create predictable localized ceramic sintering in a sample dielectric powder

1.2.3 Materials Properties Identification and Characterization

Below are the major developments under the development of materials properties identification and characterization technical area performed by the members of Code 6394 and 6350.

Selection of Ceramic Precursor Powders of Interest

In the 85GHz gyrotron system, several precursor powders were considered including yittria stabilized zirconia (YSZ), pure zirconia, and alumina. There was a lack of data concerning 85GHz coupling with these materials, so it was experimentally determined that YSZ was an appropriate precursor powder for this system. YSZ was sintered with temperatures in the 2200-2700K range, depending on powder particle size. Sintering was confirmed with electron microscope images after processing which showed necking between individual powder particles typical to conventionally sintered samples. Powders contained 7-8% yittria and had particle sizes of 30-60, 300, 1000, and 200000 nm. Both loose and compressed powder were tested. Best sintering results were achieved with 30-60 nm size powder compressed to 40-50% density pellets. For loose powder samples, a boron nitride (BN) crucible, with melt temperatures over 3000K, was used to contain the powder.

After the switch to the 2.45GHz waveguide setup, it was determined that the magneton was unable to supply sufficient power to sinter YSZ reliably. Therefore, a material with a lower sintering temperature was required and the precursor powder was changed to barium titanate (BTO) with 400 nm grain size. This powder has a coupling efficiency to 2.45GHz nearly 3x higher than YSZ, allowing for higher temperatures to be achieved while supplying less microwave power. Our best experimental results and part fabrication occurred in the 2.45GHz waveguide using lightly compressed (50% density) BTO powder within the TE_{106} microwave cavity.

Ceramic Powder Properties Literature

Measuring the dielectric properties of powders at microwave frequencies presents a variety of difficulties. The standard measurement for bulk materials at GHz frequencies is that of the resonance cavity where a precisely machined piece of material with near perfect densification and clear edges is placed inside a cavity and resonant modes are created and measured. Powder samples on the other hand have many voids within, and edges that are not well defined. This causes a number of problems for the resonance cavity, mainly because resonant mode formation depends on a clearly defined gas-solid boundary conditions. Another method for high frequency dielectric measurements is the use of micro strip lines where photolithography or a similar technique is used to create an electronic structure with a thin film or small sample volume with a firmly defined shape. Specific frequencies can then be passed across the sample and the transmission and reflection coefficients can be measured, and dielectric properties extracted. This technique does not work for powder samples because of the poorly defined shape of powder and poor electrical connection where signal is to be transferred to and from the sample. The impedance mismatch at this boundary results in untenable signal losses. This experimental problem, in addition to the lack of literature on the dielectric properties of ceramic powders, required novel property-extraction approaches described in the following subsections.

Waveguide Technique for Property Extraction

To extract the dielectric properties of ceramic precursor powders at various frequencies, densities, and temperatures, a novel transmission-line technique was used. The basis of this method is fairly straightforward for materials with low microwave absorbance, at sub-GHz frequencies, and room temperature. A solid sample of fixed and known size is placed in the path of a MHz signal and the S-parameters, complex valued transmission and reflection coefficients, are monitored. This data is then used to calculate the complex permittivity and permeability, allowing for extracting the dielectric constant and loss tangent.

For our work, a network analyzer was used to supply the microwave and analyze the S-parameters. The generated signal was confined to coaxial cables and waveguide section of known room-temperature geometry. Samples can be placed within the waveguide for the microwave to pass through. Solid samples were readily analyzed using conventional transmission-line techniques, while powder samples were contained between two pieces of PTFE of known size and shape. S-parameter measurements were conducted across 15-40 GHz and dielectric constants and loss tangents were acquired for a variety of precursor powders. Bulk samples of the precursor material were also measured and agreed with reference data. For higher temperature measurements, the waveguide can be heated, but analysis of the S-parameters increases in complexity. The mechanical deformation due to thermal expansion needs to be modeled and taken into account to modify the S-parameters prior to calculating the dielectric properties. This process was modeled up to 700 K.

Numerical Simulations and Physical Experiments

During experiments with the 2.45 GHz TE_{106} microwave cavity, several difficulties were encountered. These difficulties included the physical optimization of the microwave cavity and a curious phenomenon where sintering of precursor powder only occurs at specified locations within the cavity. In order to address these issues, an FEA model of the waveguide, crucible, and precursor powder was built. The model considered the electromagnetic microwave propagation, thermal propagation, the temperature dependent material properties, and the appropriate coupling of these three systems. This model solved the problems of optimizing the physical microwave cavity and provided an explanation of why the precursor powder was sintering in the way that it did. In short, the parallelization of the virtual and physical experiments was not only informative, but beneficial for data analysis.

Optimization of the microwave cavity is a time consuming process without the aid of numerical simulations. The process involves adjusting the ends of the cavity in order to maximize the standing wave contained in the cavity, or alternately viewed, maximize the contained power. However, local maxima exist that are orders of magnitude lower than the global maxima. These local maxima cannot be avoided without mapping the entire parameter space: tens of thousands of measurements. Additionally, the parameter map changes for each different precursor sample placed in the cavity. Instead of performing the experiment thousands of times to construct this parameter map, a virtual experiment was conducted in an empty microwave cavity with the cavity ends being adjusted. The parameter space was sampled using a Monte-Carlo method and those outputs were used as initial inputs for the virtual experiment, which ”tested” how much power was contained for each parameter input. At the location of the highest power, further optimization was done and a global maxima was located within the parameter space. This computed global maxima agreed with experimental global maxima to less than 1% difference, and required only a few hours of calculations.

Experiments within the microwave cavity resulted in precursor powder sintering at unexpected locations. Instead of sintering at locations corresponding to high electric field, it was instead localizing at different, smaller, locations along the direction of propagation. Utilizing the developed computational framework, we conducted identical experiments, and found that there is a cascading effect of interdependent feedback loops. These properties are not considered under typical electrostatic or waveguide analysis. The synopsis of this cascade is this: the microwave transfers energy to the powder, which heats up, the heat changes properties in the powder, these property changes then accelerate the microwave energy transfer. This causes only a few mm of the sample powder to sinter instead of the expected few cm as the microwave power is localized to a single place.

Once the nonlinear temperature dependent properties of the sample powder became clear, the numerical simulation framework was extended in parallel with the physical experiment, to reduce experimental time and to analyze how sample properties change with regard to T. A manual inverse problem solution was implemented via exercise of the framework by adjusting the sample properties as functions of T and matching the results from the numerical simulations to the physical experiment. This systemic approach allowed us to extract these nonlinear temperature dependent dielectric properties, which include the powder density, specific heat, dielectric constant and loss tangent.

1.2.4 Fabrication Demonstration

Below are the major developments under fabrication demonstration performed by the members of Code 6394, 6350, and 6795.

Printed Boxes

Two hollow boxes were successfully sintered using lightly compressed (50% density) BTO precursor powder and the 2.45 GHz microwave cavity system. These boxes have walls 1-2 mm in thickness to form a 20x11x8 mm hollow box of BTO. The solidifed box can be handled by hand while the surrounding and interior powder remained granular and loose. Sintering was done with ΔT of 20K/min both up and down with a 60 minute hold time at 1200 K.

A pellet of YSZ was also successfully sintered in the 85GHz gyrotron. Best sintering results were found for yittria stabilized zirconia (YSZ) when positive ΔT was less than 30K/min with rest steps of 10min at approximately 1700K and 2100K and then cooling at 10K/min to room temperature. YSZ powder was pressed to 80% density and had a more than 10% volume reduction due to sintering.

Continuum Mechanics Approach to Sintering and Application to Finite Element Analysis

Prediction of the final sintered geometry of a ceramic presents various computational challenges. To address those we developed a framework based on continuum approaches that consider the coupled non-linear thermostructural problem accounting for porosity changes based on mass conservation principles. The framework considers the momentum conservation, the inelastic material constitutive response, viscoplasticity, and sintering induced viscoplastic strain rate. The simulation framework was based on non-linear finite strain time-dependent analysis. An example application of the framework for a non-trivial geometry is shown in Fig. 2

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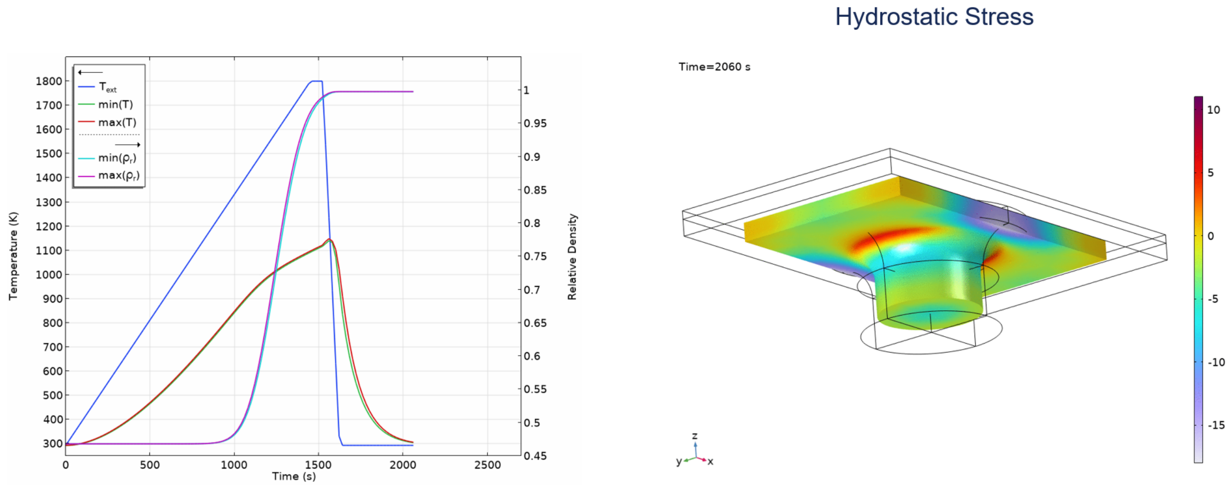

Fig. 2—Results of exercising the coupled continuuum mechanics sintering framework. Left figure: External temperature is shown in blue. Minimum and maximum temperatures inside the body of the ceramic part are shown in green and red, while porosity is shown in cyan and purple. The final density values are close to 1.0. The right figure shows the part after cool-down to room temperature and demonstrates the lvel of shrinkage and residual von Mises Stress in MPa.

1.2.5 Extra Tasks

In addition the the expected tasks, several additional tasks were accomplished by Code 6394 and 6350 as detailed below.

Hexagonal Phase BTO Found Using XRD

The microwave sintered BTO powder was further analyzed using The X-Ray Diffraction (XRD), which demonstrated a two phase mixture. Primarily, there was the expected tetragonal phase (P4mm) of typical room temperature BTO. However, a secondary phase of hexagonal BTO (P63/mmc) that usually only forms at temperatures of 1760 - 1800 K was also observed. The phase peaks which only correspond to the hexagonal peaks are notated with a red asterisk, although the phases do share other peaks as well (such as the main peak at approximately 31.3 2θ). Prior reports indicated this phase could be stabilized at room temperature by heating to 2150 K and rapidly quenched in water. The cooling rate that these samples experienced is indeed relatively fast compared to typical furnace cooled sintered samples, but since it is not equivalent to a water quench step, the hexagonal phase was only partially kinetically stabilized and resulted in the observed phase mixture.

Xray Computed MicroTomography of Fabricated Boxes

Xray Computed MicroTomography (XCMT) was used to examine the density profiles of sintered specimens from the microwave sintering process. Sections of the sintered material were scanned individually, as well as entire test specimens in their crucibles. The density maps were used to determine local sintering effectiveness and the degree of localized melting in the focused microwave beam region. Additionally, the tomography showed the degree of powder/melt motion during the experiments.

2. FUTURE PLANS AND POTENTIAL OPPORTUNITIES

This project has generated a multitude of fundamental scientific outcomes that could be benefited by additional efforts. In particular:

1. Further development at a higher TRL level will result in basic demonstartion prototypes of the technology and provide a path to the dissemination of the technology. The prototypes can be implemented both in gyrotron and magnetron systems utilizing active plasma elements or conductor/insulators assemblies.
2. The extension of the holography computational framework can result in predicting even more complex beam shaping approaches that can be tailored to various technology areas including network transmission beam shaping, medical and radar applications.
3. The sintering framework can be extended to serve as a general sintering prediction framework for sintering processes and significantly reduce the design time of complex ceramic parts. The same framework can be used to predict sintering of other times of manufacturing methods such as stereolothography based ceramic additive manufacturing.
4. The coupled multiphysis framework can be used in various hypersonic design activities to predict thermo-structural response coupled to fluid dynamics physics.

3. PUBLICATIONS

The publications produced under this project are listed below.

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