Microwave Sintering of Ceramics

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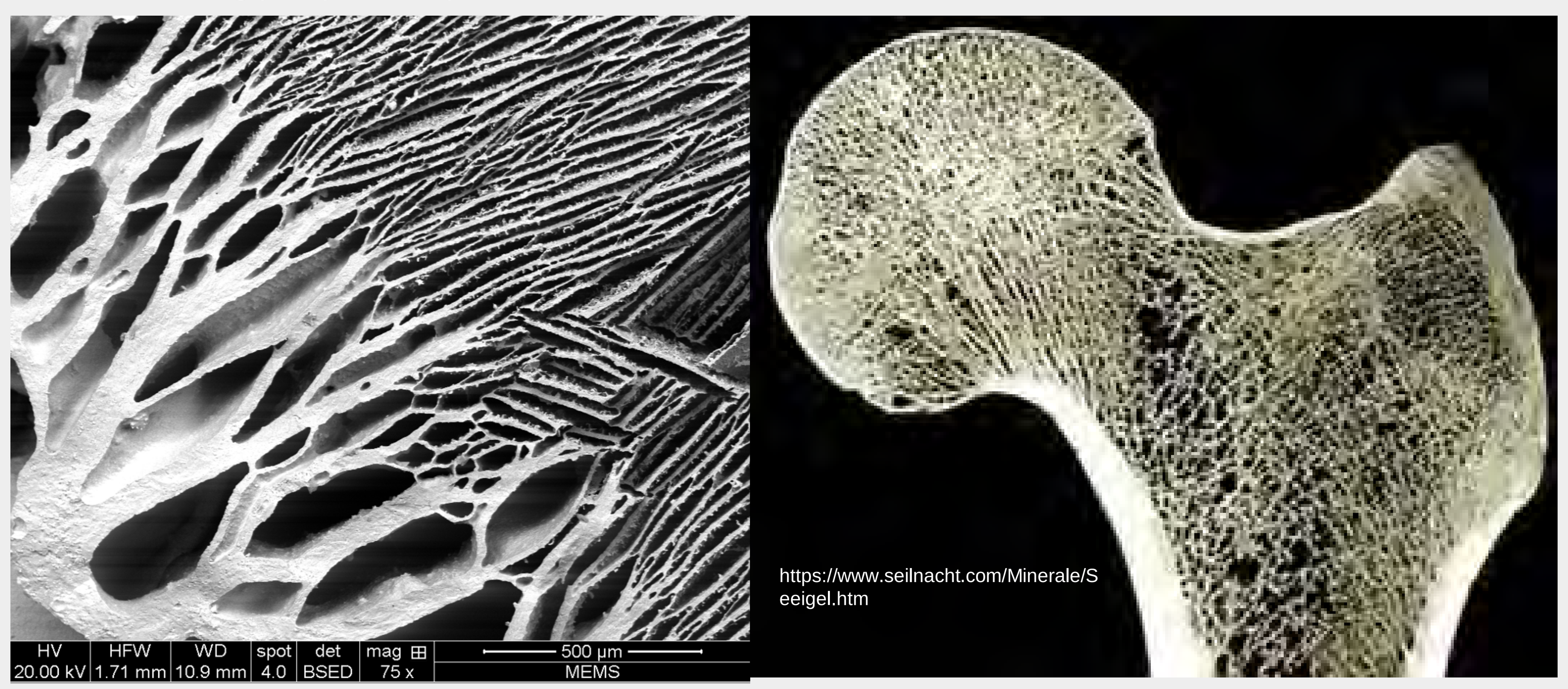
Background

The Bioinspired Science and Engineering Lab works with materials designed to mimic those found in nature. One example of this is using a biocompatible ceramic called hydroxyapatite (HA) and applying the freeze casting process, which is designed to mimic the porosity of human bones for implant purposes.

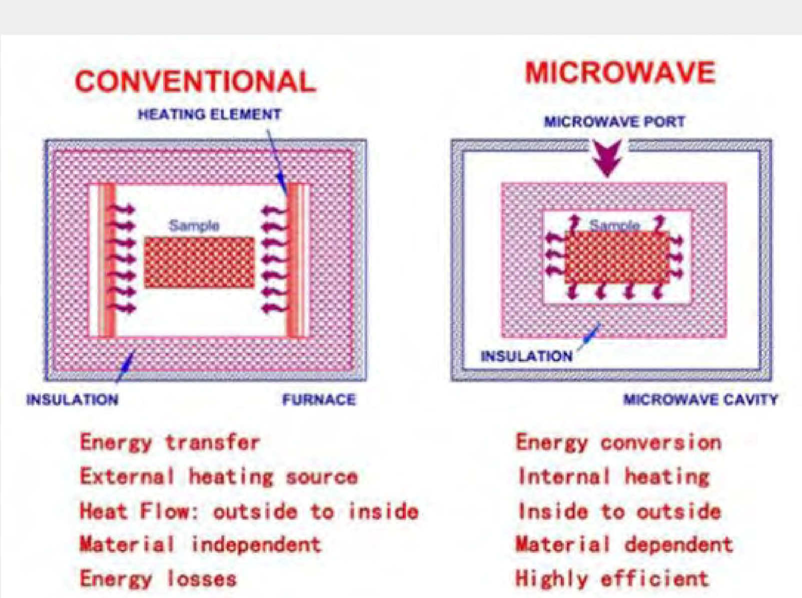
Bones have unique physical properties, and this greatly constrains how the replacement ceramics must be produced. The lab needs a way to quickly and efficiently sinter ceramics after freeze casting to produce desirable physical characteristics. Traditional sintering done in gas fired or electrically heated furnaces is energy and time intensive, thus microwave sintering was proposed since it is fast and consumes very little energy by comparison.

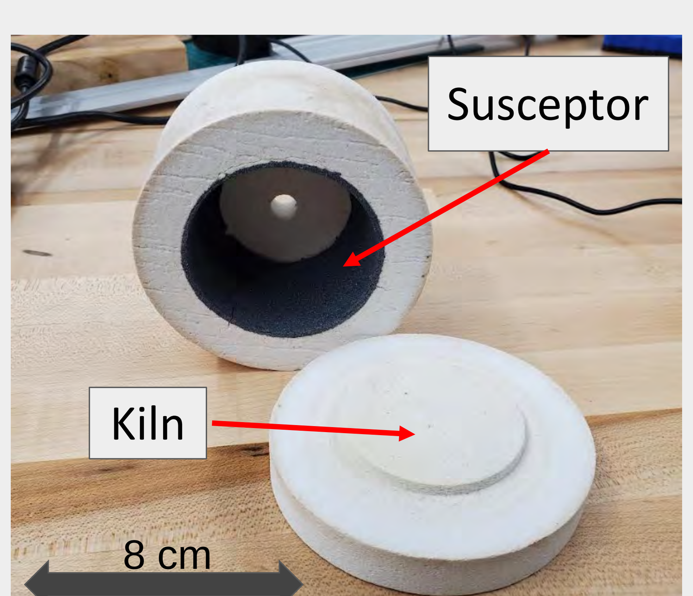


[Left] A sample of hydroxyapatite (bone substitute) sintered in the microwave furnace. Dense, small pores in combination with larger pores are some of the defining characteristics of organic bone, our test samples exhibit these traits. [Right] A cross section of human bone, showing the desired porous structure.

What is Microwave Sintering?

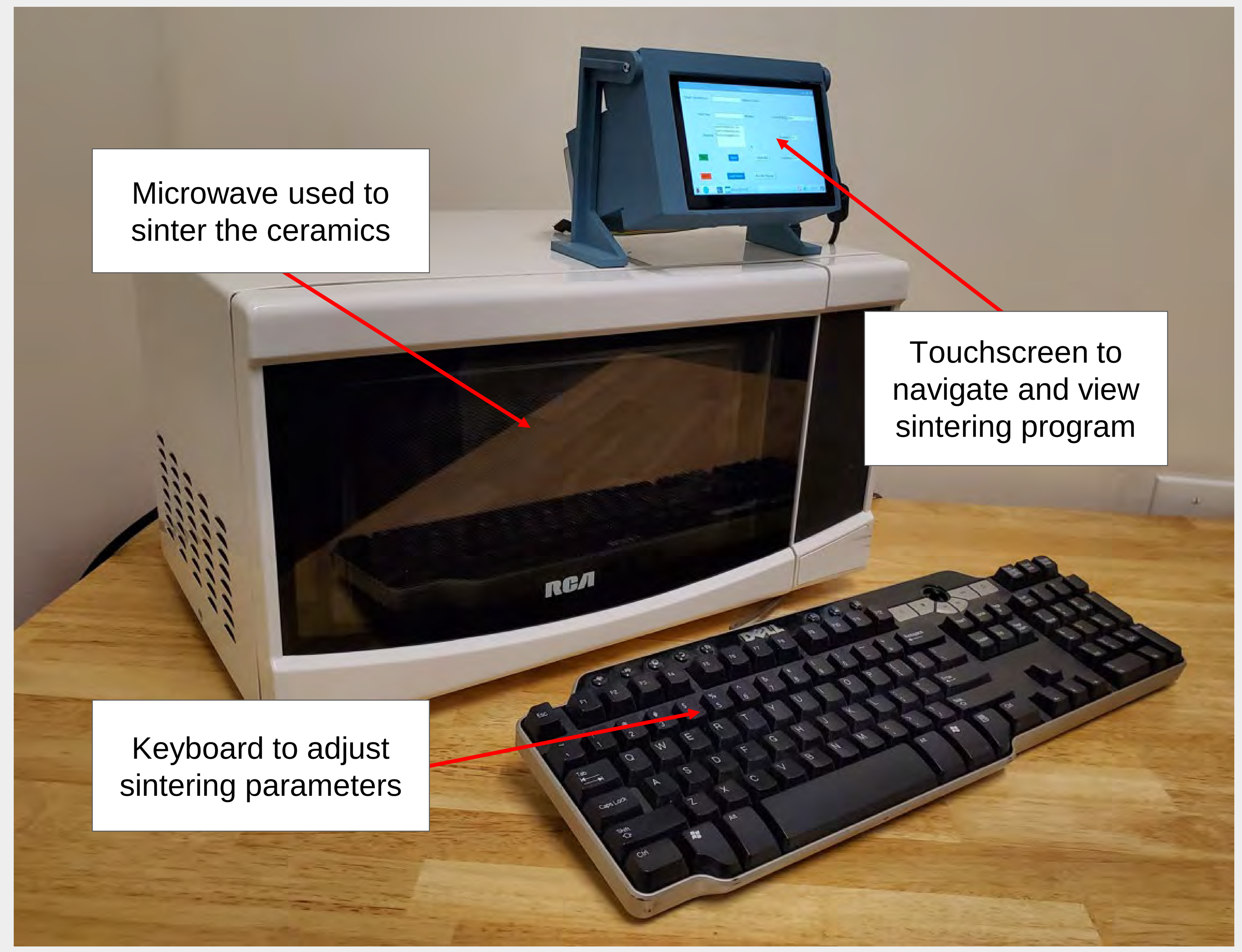
Microwave sintering is a volumetric heating process, meaning the entire ceramic piece is heated at the same time by microwave energy. A kiln is used to intercept some microwaves and heat the air around the ceramic to keep the surface and internal temperature even, the kiln also protects the user and microwave interior from the intense heat. The magnetron, which is what generates the microwaves, is highly efficient (up to 80%) at converting electrical power into microwaves that heat the ceramic, this contributes to the excellent energy efficiency of this sintering method.





https://www.langfengmetallic.com/news-in-company/the-technical-principle-of-microwave-sintering.html

Microwave Furnace Design

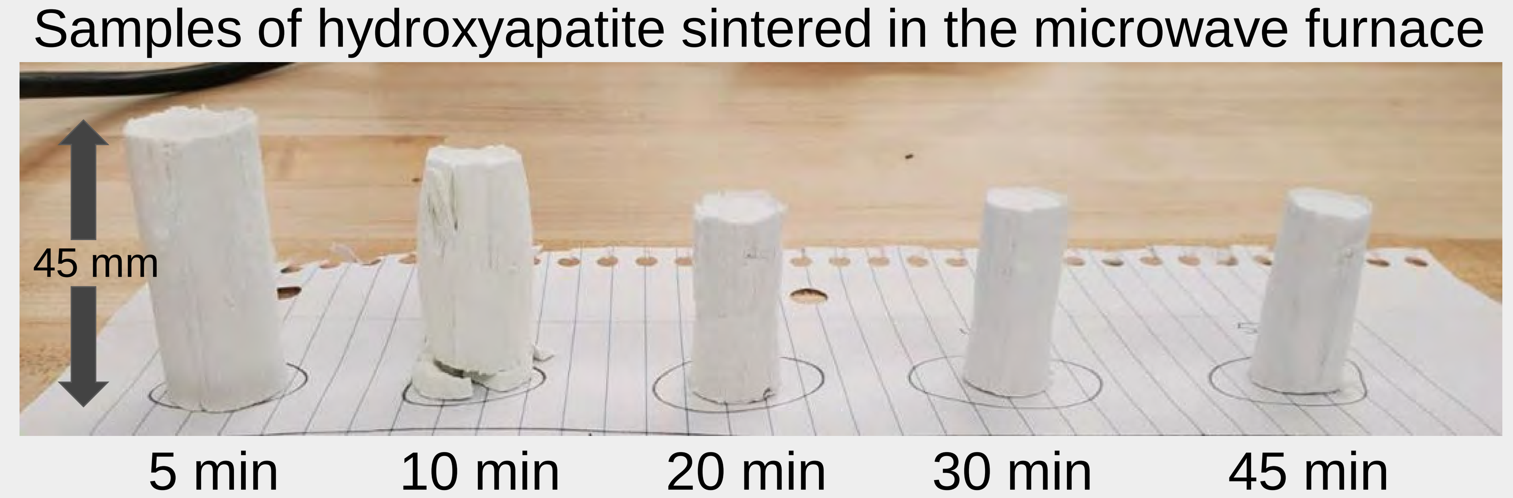


Test Methods

We conducted numerous tests on samples of HA, both previously sintered and unsintered, to assess the functionality of the furnace and to better understand the behavior of sintering materials using microwaves.

Samples were placed within the kiln, then within the microwave body, next they were heated for varying lengths of time. We measured the samples' temperature with an IR sensor before and after each test to determine the heat rate of the material. The data was then plotted against our transient heat rate model to validate it. This data will allow the Bioinspired lab to run experiments on materials and know how hot they will get and at what time they reach that temperature.

Samples of hydroxyapatite sintered in the microwave furnace

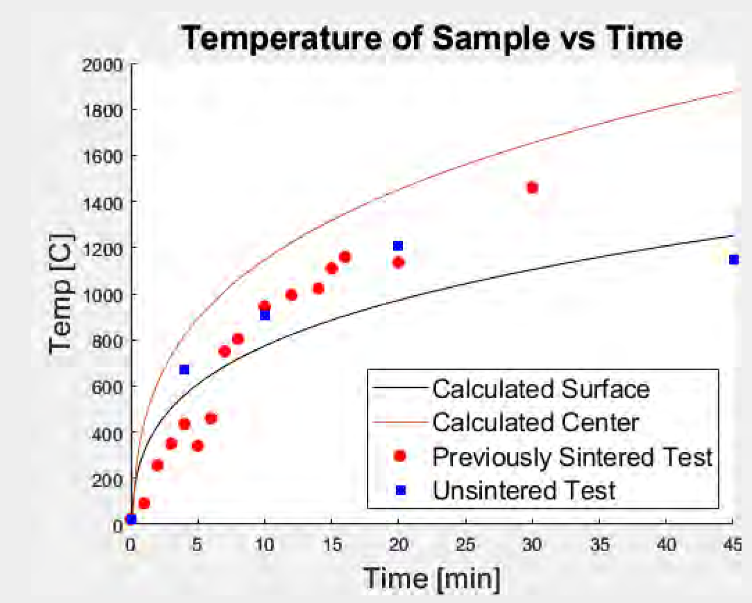


Acknowledgements

Thank you to Tony Yin and the Bioinspired Science & Engineering Lab group for providing samples of hydroxyapatite for us to test.

Thermal Model

A thermal model was produced by manipulating the transient heat conduction equation. Having an accurate thermal model allows users to predict the temperature of a sample for a given amount of time. Being able to predict the temperature of ceramic samples can significantly reduce time spent characterizing a material.



$$\frac{T_s - \alpha\tau}{T_i - \alpha\tau} = e^{\frac{t}{\tau}},$$

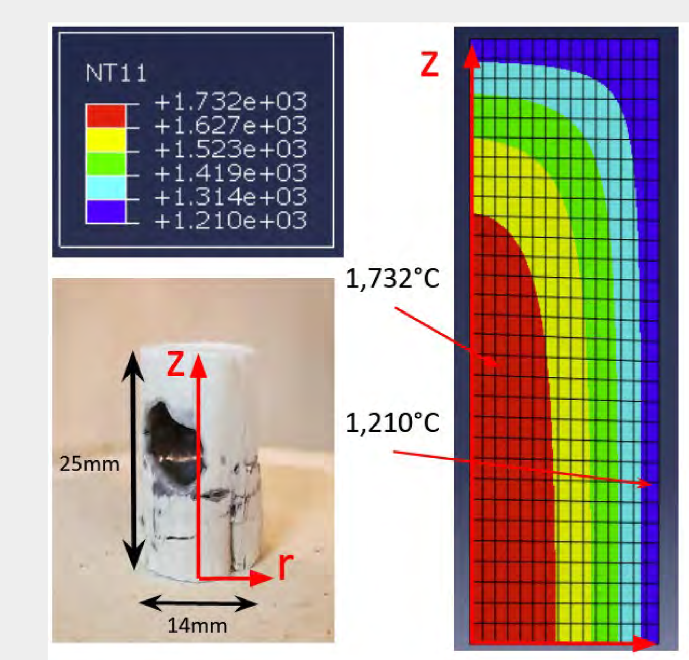
$$\alpha = \frac{q_{microwave}}{\rho \cdot c_v},$$

$$\frac{1}{\tau} = \frac{4\sigma \cdot (T_{avg})^3}{R_{rad}} + R_{conv},$$

T_s = (susceptor - material) temp. difference
T_i = initial temp. difference
$q_{microwave}$ = radiation from microwave
σ = Stefan Boltzmann constant
T_{avg} = average between susceptor and material temp

FEA

To predict the temperature at the center of our samples, given a known surface temperature from testing, we developed an FEA model of the ceramic to estimate the maximum temperature in the sample. Boundary conditions were obtained from testing and research. This analysis did not account for the susceptor absorbing some of the radiation. These results demonstrate the nonuniform heating.



Conclusion

Overall, the microwave sintering furnace is successful as it's able to reach sintering surface temperatures of 1200 °C, enough to melt the center of HA. The furnace is a compact and simple to use device that take minutes to sinter ceramics rather than the hours of a conventional furnace. From the thermal model and the FEA analysis we can predict how long to sinter the ceramics and have a good understanding of how to avoid melting the center.

Table 1: A table summarizing the project objectives.

#	Metric	Unit	Goal Value	Obtained Value
1	Maximum temperature	°C	1500	1400
2	Heat rate	°C/min	5<HR<100	60
3	Accurate temperature measurement	°C	±5% of target	±2.5%
4	Hold time	min	1-10	NA
5	Sinter ceramic	binary	1	1
6	Maximum external microwave temperature	°C	<150	160
7	Internal cavity volume	cm^3	>164	NA
8	Number of samples in kiln	#	>=1	1