

Microwave Hybrid Processing of Hydroxyapatite

Ehsani N¹, Sorrell CC² and Ruys AJ^{3*}

¹Faculty of Materials and Manufacturing Technology, MUT University of Technology, Tehran 84817-75631, Iran

²Graduate School for Biomedical Engineering, University of New South Wales, Sydney, NSW 2052, Australia

³Centre for Advanced Materials Technology, Department of Mechanical and Mechatronic Engineering, University of Sydney, Sydney, NSW, 2006, Australia

Abstract

The effects of microwave hybrid heating on the densification and decomposition behaviour of four commercially available pre-calcined HAp powders (Plasma Biotol P81, P88, P120 and P149) and a bead milled HAp powder (BM16) were studied. The results were compared with conventionally sintered samples. Samples microwave heated to 1100°C for 10 min at 10,000°C.h⁻¹ were 15 to 40% denser than the samples conventionally heated at same temperature for 60 min at 300°C.h⁻¹. Microwave heating reduced densification temperatures and soak times by as much as ~150°C-200°C and ~57 min, respectively. Overall, the microwave sintering cycle was 20-30 times faster.

Introduction

Hydroxyapatite [HAp] clearly has a great potential for biomedical applications because of its similarity in chemical composition and crystallographic structure to human hard tissue [1,2]. However, relatively poor strength and fracture toughness have restricted its extensive applications. A great deal of work has been done on the research and development of HAp ceramics as biomaterials [3-7]. One possible solution for improving the mechanical properties is microwave sintering of HAp [8-11].

Microwave sintering of HAp has been studied by Fang et al. [12-14] and Agrawal et al. [15]. They successfully made a number of transparent, white and porous HAp ceramics. Commercial microwave furnaces (500 and 1000 W) with cylindrical ZrO₂ or rod-like SiC susceptors were used. Fully dense (100%) and transparent hydroxyapatite was produced by microwave sintering at 1100°C-1175°C [15], whereas conventionally produced samples from the same powder did not show complete transparency.

Microwave hybrid heating [MHH]

Microwaves form the part of the electromagnetic spectrum between radio waves and infrared light, occupying the frequency range 0.3 to 300 GHz, with the corresponding wave length range of 1 mm to 1 m [16]. Allocated frequencies for industrial, scientific and medical use are shown in Figure 1. Among these frequencies, only 9.15 MHz and 2.45 GHz are commonly used in industrial equipment [17]. Microwave heating results from the interaction between the microwaves and the

material that is being heated, resulting in energy dissipation by various mechanisms [18,19].

Microwave hybrid heating is a unique combination of conventional and microwave heating techniques, developed specifically for materials with low loss factor and low thermal conductivity [20,21]. Significant microwave heating problems can be solved by this novel technique [22-25]. In the MHH technique, a strongly microwave absorbent material is used as a susceptor to raise the temperature of a weakly microwave absorbent sample above the critical temperature (T_{crit}). Once this temperature is reached, the sample can directly absorb microwave radiation. Therefore, at low temperatures the susceptor absorbs microwave energy and transmits heat into the sample by conventional heat transfer mechanisms, primarily radiation [20,26]. This technique facilitates the attainment of very high heating rates in a 2.45 GHz multimode microwave cavity [26]. Good temperature homogeneity can be achieved in large samples during MHH. MHH ceramics have shown greater microstructural uniformity and superior mechanical properties compared with conventional sintering using similar heating cycles.

The most common susceptor materials are SiC and ZrO₂. Y_xNi_{1-x}O compositions have recently been studied as a new susceptor material for high temperatures and oxidising conditions by Komarenko et al. [27]. Only pure NiO was found suitable as a microwave susceptor.

Powder	Surface Area (m ² .g)	Particle Size (µm)
P81	19.9	6.97
P88	20.5	3.16
P120	41.3	1.84
P149	18.6	5.10
BM16	85.2	0.48

Table 1: Characteristics of HAp powders.

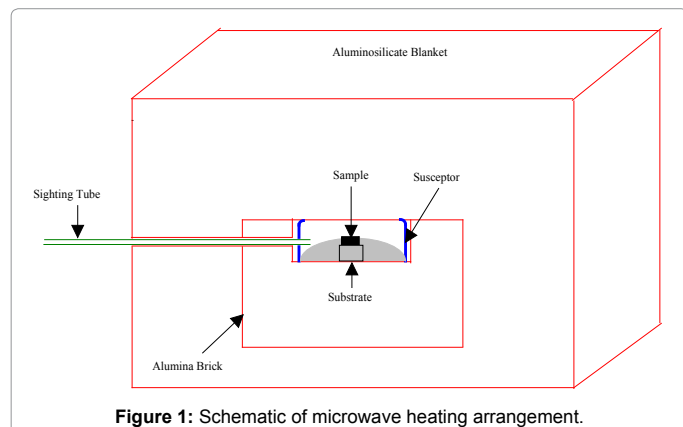


Figure 1: Schematic of microwave heating arrangement.

*Corresponding author: Ruys AJ, Centre for Advanced Materials Technology, Department of Mechanical and Mechatronic Engineering, University of Sydney, Sydney, NSW, 2006, Australia, E-mail: Andrew.ruys@sydney.edu.au

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Experimental Procedure

Raw materials

Four commercially available pre-calcined HAp powders, including P81, P88, P120 and P149 (Plasma Biotol Ltd., Tideswell, Derbyshire, England), were used. BM16 powder was prepared by bead milling P149 HAp powder, with sixteen passes. Table 1 shows the average particle size and specific surface area of the HAp powders used in this work. For sintering studies, the powders were uniaxially pressed into pellets of 12.7 mm diameter.

Microwave hybrid heating: A 1.5 kW, 2.45 GHz variable-power microwave furnace (Ceramic Engineering, Sydney, Australia), with proportional power control (amplitude variation) and built-in mode stirrer, was used at the 65% power level for all experiments.

HAp is only weakly microwave-absorbent at low temperatures. It therefore cannot be heated from room temperature without a susceptor. However, like most ceramics, its microwave absorption capability increases with temperature [28]. Therefore, a microwave hybrid heating procedure was required to heat the HAp samples from room temperature to the critical temperature (T_{crit}), above which HAp can efficiently absorb microwave radiation cylindrical ZrO₂ or rod-like SiC susceptors.

A cylindrical clay-bonded silicon carbide susceptor ($\varnothing=60$ mm, $h=20$ mm) was positioned around the sample. The susceptor was placed inside an alumina brick ($120 \times 20 \times 80$ mm), which was surrounded by a layer ($240 \times 240 \times 200$ mm) of fibrous aluminosilicate insulation (Kaowool, Morganite Industrial Products, Sydney, Australia). This arrangement is shown schematically in Figure 1. For each experimental run, one sample was placed at the centre of the susceptor. The temperature was measured with an infrared pyrometer, which was calibrated during the cool-down cycle using a Pt - Pt13Rh thermocouple. A standard heating rate of $\sim 10,000^{\circ}\text{C}\cdot\text{h}^{-1}$ and soak time of 10 min were used. The mode stirrer was used at all times to produce a more uniform microwave field distribution across the cavity.

Conventional heating: An electric furnace ($300 \times 200 \times 120$ mm) with SiC elements (Gallenkamp, London, UK) was used for the conventional heating of the control samples. A standard heating rate of $300^{\circ}\text{C}\cdot\text{h}^{-1}$ (the furnace maximum) and soak time of 60 min were used in all cases. No serious overshooting was observed by such a high heating rate. Two sets of experimental trials were carried out.

Comparative densification and decomposition behaviour: Pellets from each of the HAp powders were sintered at a range of temperatures from 900°C to 1400°C in 100°C steps by microwave and conventional heating. After sintering, the apparent density and %HAp yield of the pellets were measured according to the methods described by Ruys et al. [29].

Effect of microwave soak time: To investigate the effect of microwave soak time, one pellet from each powder was microwave sintered at 1100°C using soak times of 1, 3, 10 and 30 min. No conventionally sintered controls were used in this study. After sintering, the apparent density of the pellets was measured to determine the effects of microwave soak time on the densification behaviour of HAp ceramics.

Results and Discussion

Comparative densification behaviour

Figures 2-6 present the sintering curves of the HAp powders

sintered by microwave and conventional heating. They reveal that, with microwave sintering, densification began at $\sim 900^{\circ}\text{C}$ - 1000°C , which was $\sim 100^{\circ}\text{C}$ - 150°C lower than for conventional sintering. These data show that the maximum densities were achieved at $\sim 1200^{\circ}\text{C}$ - 1400°C (the sintering plateau temperature for microwave heating) for microwave heating as compared with $\sim 1300^{\circ}\text{C}$ - 1400°C (the sintering plateau temperature for conventional heating) for conventional heating. In other words, microwave heating below the conventional heating plateau temperature significantly improved densification. However, the densities obtained above the conventional heating plateau temperature were similar for microwave or conventional heating.

The densities attained after sintering at 1100°C using microwave and conventional sintering are shown in Figure 7. These data show

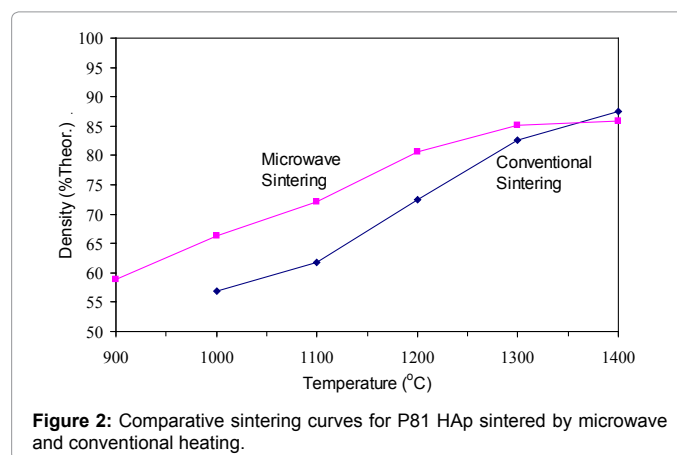


Figure 2: Comparative sintering curves for P81 HAp sintered by microwave and conventional heating.

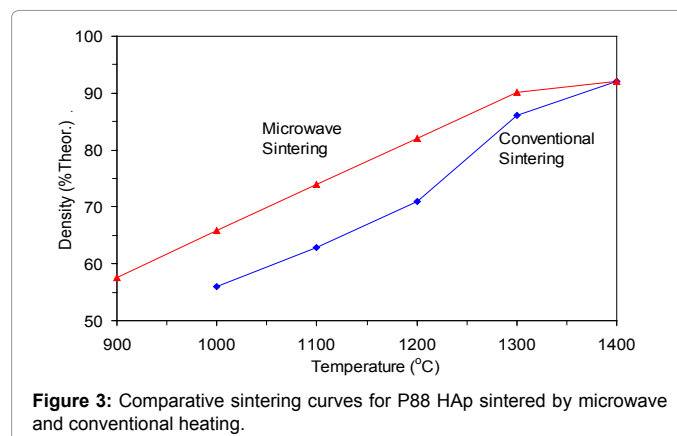


Figure 3: Comparative sintering curves for P88 HAp sintered by microwave and conventional heating.

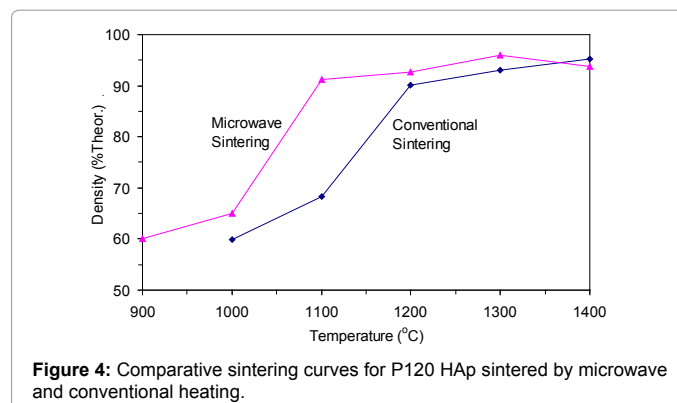


Figure 4: Comparative sintering curves for P120 HAp sintered by microwave and conventional heating.

that microwave heating significantly improved densification for each of the powders trialled. Overall, the improvement in densification by microwave heating at 1100°C was of the order of ~11-30%. Although the soaking time for the microwave and conventionally processed samples are different, the comparison is valid below the composition temperature, where no decomposition occurs. At higher temperature, however, using identical soaking times would be more reliable.

Therefore the main difference between microwave and conventional heating in this trial was the fact that densification occurred at lower temperatures for the former. This is advantageous in the case of HAP since lower sintering temperatures reduce the risk of decomposition. Therefore, it appears that microwave sintering offers a solution to the problem of trying to achieve maximal densification of HAP without the decomposition.

Enhanced densification, and lower sintering temperatures compared to conventional heating have already been reported for microwave heating of ceramics [30-40]. The mechanisms and reasons for the enhanced sintering of ceramics by microwave heating are not completely understood [41]. However the following mechanisms are proposed:

High diffusion rate: It has been suggested that the lower sintering temperatures and the higher sintering rates during microwave heating are caused by enhanced diffusion rates induced by the electric field [42-45]. The diffusion rate enhancement by microwaves has been reported for several materials [44-48]. It has also been found that the diffusion rates were dependent, at least in part, on the input power level. Janney and Kimery [41] found that the activation energy for diffusion of ^{18}O in sapphire was 410 kJ/mol for microwave heating and 710 kJ/mol for conventional heating. The presence of a microwave effect in the single crystal suggests that there would be a significant direct interaction between the crystals in polycrystalline ceramics and microwaves, and so microwave coupling cannot be attributed solely to grain boundary effects.

Preferential grain boundary coupling: Grain boundary coupling could have an important effect on the microwave sintering of ceramics. It has been suggested that the enhanced densification of ceramics by microwave sintering could be caused by accelerated diffusion rates within the grain boundaries [49]. This would be caused by preferential interaction of the microwaves with the interfaces, leading to higher temperatures in these regions. Several different mechanisms have been proposed for this temperature heterogeneity.

As previously have been suggested by Booske et al. [49,50], the loss tangent of the grain boundaries is higher than within the grains, and consequently microwave absorption and the temperature are higher at the grain boundaries. The electric field intensity in the areas of low

dielectric constant is higher than that in the areas of high dielectric constant [51]. Since the dielectric constant of the pores at the grain boundaries ($\epsilon'_{\text{air}}=0.1$) is much lower than the dielectric constant of the ceramic grains ($\epsilon'_{\text{alumina}}=0.9$), the electric field intensity between grains is much higher. Therefore, the field strength (E) and temperature are greater at the grain boundaries.

Another possible mechanism is that microwave radiation might generate dielectric breakdown at the mutual contact points of the green ceramic particles [52,53]. This phenomenon, which is common in polycrystalline high-dielectric constant materials, can lead to temperature gradients between the surface and centre of the grains. Meek et al. [54] found that the structure of the surface and the centre of the particles were crystalline in conventionally heated samples. However, in the microwave-heated samples, the structure of the centre

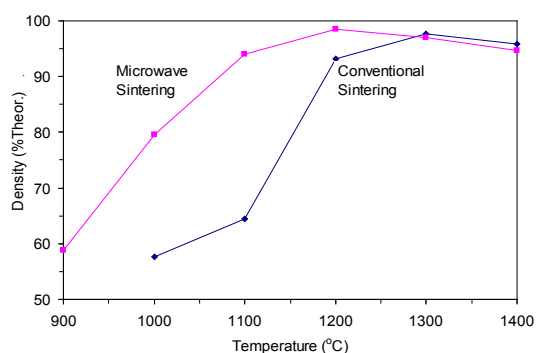


Figure 6: Comparative sintering curves for BM16 HAP sintered by microwave and conventional heating.

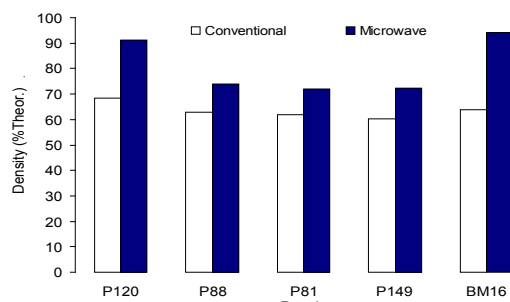


Figure 7: Comparative densities for HAP sintered at 1100°C.

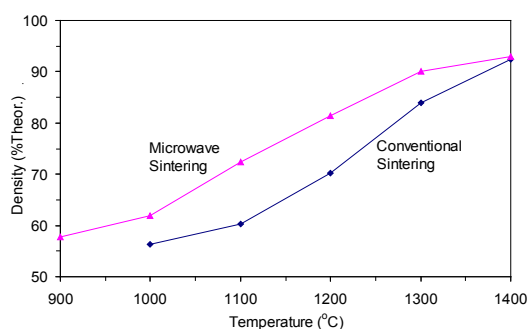


Figure 5: Comparative sintering curves for P149 HAP sintered by microwave and conventional heating.

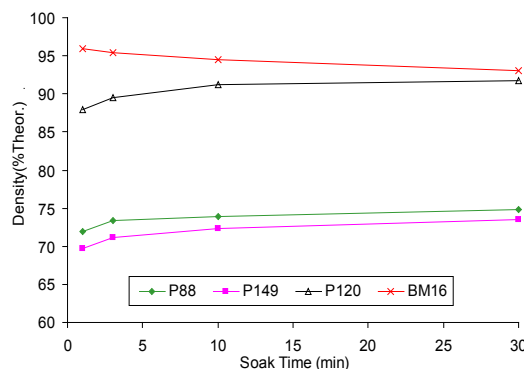


Figure 8: HAP density versus microwave soak time.

was crystalline while the structure of the surface was amorphous. The existence of an amorphous layer on the surface of the microwave sintered grains suggests the presence of a liquid phase at the particle surfaces during microwave sintering. This would enhance densification.

It has also been suggested that, in microwave sintering, the temperature between particles is higher than the apparent or measured temperature and the supposed formation temperature of the liquid phase [55]. This temperature heterogeneity will decrease the liquid viscosity in the grain boundaries. Moreover, the vibration rate and amplitude of the liquid molecules will be increased by preferential coupling of the microwaves with the liquid formed in the grain boundaries. This also will reduce the liquid viscosity. The presence of a liquid with low viscosity increases the rate of grain rearrangement, thereby enhancing the densification rate.

This theory has been confirmed by Tiegs et al. [56-59], who have shown that preferential coupling of microwaves with the grain boundaries in microwave heated Si_3N_4 -based ceramics leads to improved densification. Ferber et al. [60] established that the improved creep resistance of microwave-heated (Si_3N_4 -6 Y_2O_3 -2 Al_2O_3) was due to the preferential coupling of microwave radiation with the intergranular phases. It has been suggested that relatively small temperature gradients across the grains can induce a driving force to encourage densification [54].

Figures 3-7 shows that the temperature for the onset of densification for either microwave or conventional heating was lower for the powders with smaller particle size and higher surface area (such as BM16 and P120). The sintering characteristics of a powder depend on the surface area, particle size, particle size distribution, morphology and reactivity [61]. For a given chemical reactivity, densification at a given temperature will be enhanced with a finer particle size, higher surface area, and appropriate size distribution for dense particle packing. The temperature at which the maximum density was achieved also depended on the particle size and surface area of the powders. In case of BM16 impurities also have possibly affected the density. It was lower for the powders with a smaller particle size and higher surface area.

Figure 7 shows that the densification enhancement of microwave heating was strongly dependent on the particle size of the powders. The densification enhancement for the fine HAp powders, such as BM16 and P120, was much greater than that for the coarser HAp powders, such as P81, P88 and P149. Compared with conventional sintering, microwave sintering of fine powder (BM16) at 1100°C led to a ~30% (64.5% versus

94.5% dense) densification enhancement whereas it led to only a ~11% increase in densification of coarse powder (62.8% versus 73.9% dense). This enhanced effect with finer powders suggests that the particle size of the powders and, by association, the grain boundary area strongly influenced the degree of microwave densification. These accords with the theories discussed previously, where preferential coupling of grain boundaries with microwaves is one of the densification enhancement mechanisms of microwave sintering [49,51]. Since the fine powders have a higher surface area and higher grain boundary area, they are more susceptible to preferential grain boundary coupling.

Comparative decomposition behaviour: The %HAp yields of the samples sintered by microwave and conventional heating are shown in Table 2. The data reveal that the decomposition effect was slightly higher for microwave sintering than for conventional sintering. HAp decomposition is a diffusion controlled mechanism [62]. Since microwave heating presumably enhances diffusion rates [44-56], it is possible that it could enhance HAp decomposition. However, the data suggest that the effect of microwave heating on densification was much more significant than that on decomposition.

Tables 2 and Table 3, and Figure 6 show that, compared with conventional sintering, microwave sintering of BM16 powder at 1100°C led to a ~30% (64.5% versus 94.5% dense) densification enhancement whereas, it led to only a 5% increase in decomposition (76% versus 81%). The fact that densification is enhanced much more than decomposition is a positive result, which points to practical benefits in processing of reinforced HAp biomaterials. The reason for this strong enhancement of densification (compared with a weak enhancement of decomposition) is that the decomposition rate during microwave heating was probably increased by the induced higher diffusion rate, whereas densification was enhanced by both the induced higher diffusion rate and the preferential grain boundary coupling mechanism. Therefore, any disadvantage in decomposition should be partially compensated by advantages in densification.

Effect of microwave soak time: A number of combinations of powder type and soak time were trialled at a soak temperature of 1100°C. The results are shown in Figure 8. These data indicate that, for three pure HAp powders (P88, P120 and P149), increasing soak times led to a slight improvement in densification level, although little improvement occurred for soak times above 3 to 10 min. Therefore, a 3 min soak time at 1100°C was all that was required for effective densification. This

TEMP (°C)	900°C		1000°C		1100°C		1200°C		1300°C		1400°C	
	CONV	MICRO	CONV	MICRO	CONV	MICRO	CONV	MICRO	CONV	MICRO	CONV	MICRO
P120		100	100	100	100	100	100	100	100	100	100	100
P88		100	100	100	100	100	100	100	100	100	100	91.0
P81		100	100	100	100	100	100	100	100	100	100	88.0
P149		100	100	100	100	100	100	100	100	100	100	83.0
BM16		85.0	83.0	75.0	81.0	76.0	78.0	68.5	70.0	66.0	63.0	61.0

Table 2: Comparative %HAp yield achieved by conventional and microwave sintering.

POWDER TEMP (°C)	P120		P88		P81		P149		M16	
	CONV	MICRO	CONV	MICRO	CONV	MICRO	CONV	MICRO	CONV	MICRO
900		60.1		57.5		58.9		57.8		58.9
1000	59.8	65.0	56.1	65.8	56.9	66.4	56.4	62.0	57.7	79.5
1100	68.3	91.2	62.8	73.9	61.7	72.1	60.2	72.4	64.5	94.5
1200	90.1	92.6	70.9	82.0	72.5	80.6	70.1	81.4	93.1	98.4
1300	93.1	95.9	86.1	90.1	82.6	85.2	83.9	82.0	97.7	96.9
1400	94.2	93.8	92.2	92.2	87.5	85.8	92.4	87.9	95.9	94.7

Table 3: Comparative density of HAp powders sintered by conventional and microwave heating.

suggests that increasing the soak time at temperatures below T_{onset} will increase the density only marginally.

However, for the contaminated HAp powder (BM16), longer soak times reduced the density. This was probably owing to decomposition effect of impurities since the data in Table 2 shows that the decomposition temperature (T_{onset}) of BM16 powder was much lower than 1100°C. Since decomposition is a time-dependent phenomenon [63], longer soak times led to more decomposition, which aerated the microstructure and prevented densification.

This was confirmed by the decomposition data, which show that the %HAp yield decreased with respect to soak time from 82.5% (1 min soak) to 78.9% (3 min), 76.3% (10 min) and 72.1% (30 min). Therefore, it can be assumed that increasing soak times will increase the density only at temperatures below T_{onset} . Excessive soak times at temperatures above T_{onset} will in fact be detrimental and decrease density through decomposition effects.

Conclusions

1. Samples microwave-heated to 1100°C at 10,000°C.h⁻¹ were ~11 to 30% denser than the samples conventionally heated at the same temperature for 60 min at 300°C.h⁻¹.

2. With microwave heating, densification temperatures were ~100°C-150°C lower than for conventional heating. The lower densification temperature for microwave heating offers a practical solution to the problem of HAp decomposition before attaining maximum sintered density.

3. Enhanced densification and lower sintering temperatures compared to conventional heating are assumed to be due to a) enhanced diffusion rates induced by the electric field and b) preferential grain boundary coupling with microwave radiation.

4. Owing to the preferential grain boundary coupling mechanism, densification enhancement by microwave heating (compared with conventional heating) was more pronounced when the powder was finer. It was ~30% for the fine powder (BM16) and ~11% for the coarse powder (p88).

5. The %HAp yields of the samples sintered by microwave and conventional heating were slightly lower for microwave sintering than for conventional sintering. However, the effect of microwave heating on densification was much more significant than that on decomposition.

6. The optimal microwave soak time at 1100°C was ~3 min for a heating rate of 10,000°C/h. Longer soak times at temperatures below T_{onset} led to little densification improvement. Longer soak times above T_{onset} instead led to more decomposition, which prevented densification.

7. Overall, the microwave sintering cycle was 20-30 times faster than for conventional sintering.

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