JOURNAL OF MECHANICAL ENGINEERING AND SCIENCES (JMES)
ISSN: 2289-4659 e-ISSN: 2231-8380
VOL. 14, ISSUE 2, 6770 – 6788
DOI: https://doi.org/10.15282/jmes.14.2.2020.18.0530

ORIGINAL ARTICLE

Size-effect in microwave processing of engineering materials - A review

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ABSTRACT – The size of material units is especially critical in manufacturing processes where thermal energy interacts with the material. The microwave energy is widely used to process the materials in industries such as food processing, chemical, manufacturing etc. due to its unique heating characteristics. In microwave processing, energy is generated and absorbed inside the material during irradiation. The energy absorbed per unit volume of the material depends upon its size. The smaller size candidate materials have more effective surface area to absorb microwave energy than the bulk ones and usually yield lesser defects. This review paper summarizes the fundamentals of size-effect, microwave–materials interaction and input/output parameters in microwave material processing. Further, size-effect in microwave processing of different type of engineering materials (metal based, ceramic based and polymer based) have been discussed in terms of energy absorption and improvement in product attributes. The challenges in microwave processing of metal based materials have been identified and opportunities have been outlined in order to improve the properties vis-à-vis particle sizes during microwave processing.

ARTICLE HISTORY
Revised: 3rd Sept 2019
Accepted: 23rd Apr 2020

KEYWORDS
Size-effect;
microwave processing;
metal;
ceramic;
polymer.

INTRODUCTION

Generally, poor material properties and higher volume fraction of flaws in a product are result of poor raw material selection, poor process control and limitations of the manufacturing processes. Better raw material selection results in better product quality; however, process control and improvement in manufacturing processes remain critical consequently, it has been a continued endeavor either to improve the existing processes or to develop advanced techniques for material processing. In the past few years, microwave energy has been increasingly used to process different materials for application like food processing [1, 2] chemical synthesis [3] drying [4], heating [5–12] melting [13], sintering [14–56], coating [57–60], joining [61–74], cladding [75–82], casting [83–86] and curing [87–100] etc. Now a days, some of the microwave processing techniques are gaining fast popularity in material processing industries due to energy saving, eco-friendly, rapid and uniform heating characteristics of microwave energy [101–107]. Use of microwave energy in manufacturing industries leading to improvement in the properties of the processed materials compare to conventional thermal heating [102]. However, many microwave processing techniques such as joining, cladding, casting etc. are still in the developing phase and need to be industrialized with further understanding [101–103]. Significantly, size of the target materials plays an important role during microwave processing and in controlling the properties of the processed material. Overall, the size of constituent material in a product has vital influence on it's mechanical and physical properties [16–26, 52–56, 75–82]. This effect was first speculated by Leonardo-da-Vinci in the year 1500. He observed that the strength of a cord increases as its length decreases [108]. Latter, this effect was observed and reported by different scientists/philosophers in terms of material properties, flaws and length of materials [108]. Some of the notable observations have been shown in Figure 1. The reported effects of material size in various applications of microwave processing have been illustrated schematically in Figure 2. It is interesting to note that although the size-effect phenomenon could catch the attention of the scientists long ago, serious research concerning the possible consequences and remedial measures did not get due attention till twentieth century.

In the field of microwave processing of engineering materials, for example metal powders, polymer, ceramic and their composites have been reported considerably, however; only a few authors have reported bulk metals processing. Better mechanical and metallurgical properties such as higher densification rate, density, ultimate tensile strength, microhardness and lower sintering temperatures were reported for microwave sintering of most of the metal powders (pure or alloy) size reaches to nano range [14, 15, 26–29, 37, 48, 52–56] except aluminium [6] using lower diameter powder particles. Al is a conductive in nature and highly passivated with an aluminium oxide (Al_2O_3). Smaller particle size of Al shows higher percentage of Al_2O_3 which increases the electrical resistance between the particles due to formation of thicker oxide shell than the larger Al particles. The thicker oxide shell (8.73 nm) resulting in lower heating of the nano/submicron size Al particles. It has been reported that smaller size reinforcements and matrix in metal matrix composites (MMC) improve mechanical and metallurgical properties of MMCs due to localize susceptor heating of ceramics reinforcements [17–25]. Satisfactory joint efficiency, mechanical and metallurgical properties of joints was

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reported in microwave joining of bulk metals with smaller size filler metal powders [61, 62, 67–70]. Nano powders provide better micro-hardness and wear resistance in clad than powders in micro scale used in microwave cladding of metallic substrates[75–78, 80–82].

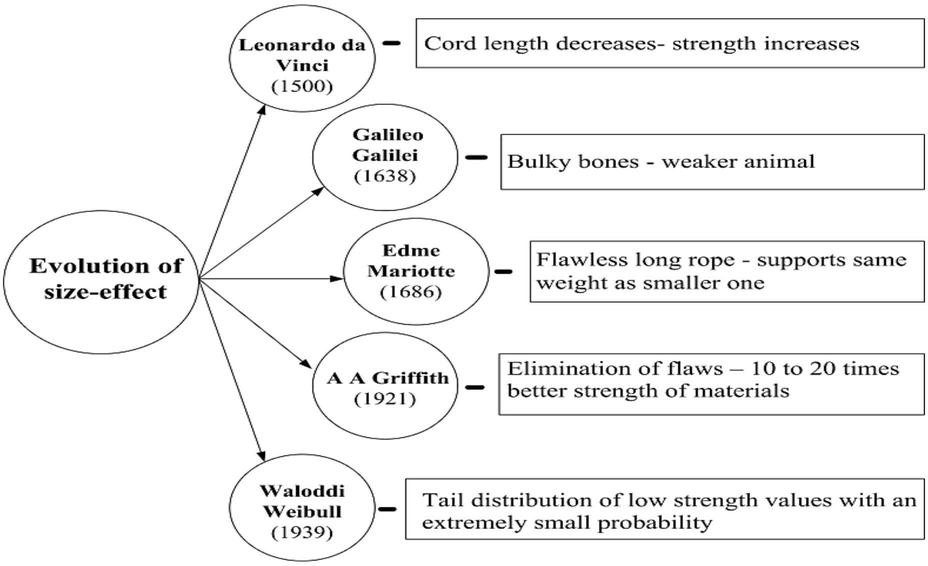

Figure 1. Evolution of size effect concept.

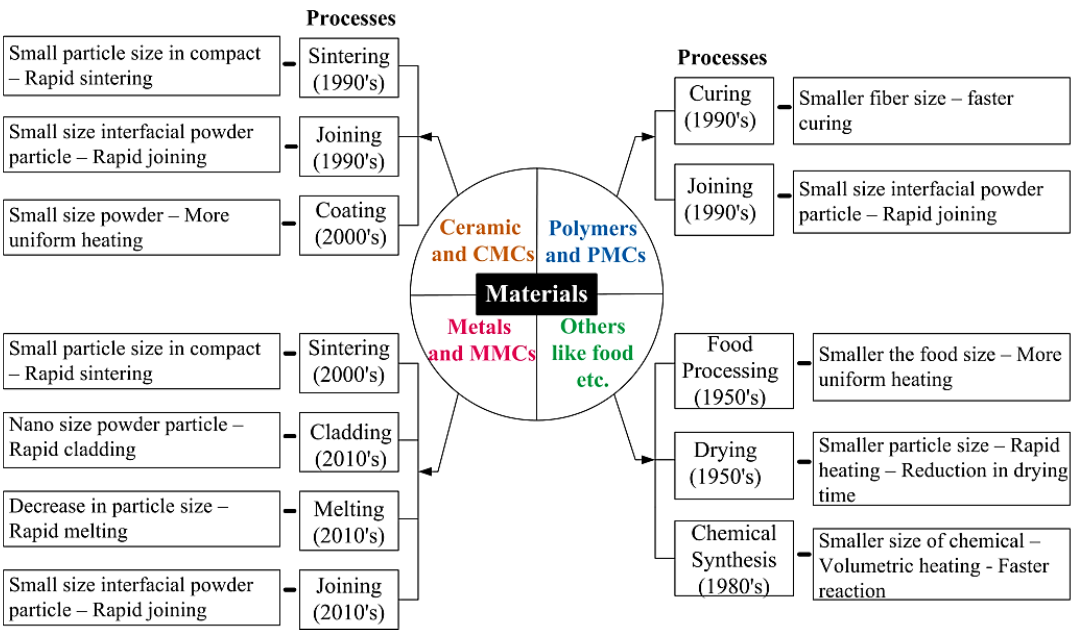

Figure 2. Effects of material size in various applications of microwave processing.

Microwave sintering of ceramics were reported with better mechanical properties due to high densification rate and more uniform heating. Increase in mechanical properties of ceramic matrix composites with smaller reinforcement size were reported [43–47, 49–51]. Better curing and increase in conductivity channels associated with smaller size reinforcements in Polymer Matrix Composites (PMC) were also reported [87, 88, 93–97]. The metallic fillers in nano range provide conductive PMCs; better mechanical properties were reported [89–92, 99, 100]. This paper reports on size-effect in microwave processing of engineering materials for example polymers, ceramics, metals and their composites. The energy absorption due to change is size have been discussed in these materials. The challenges in the field of microwave processing of metallic materials have been discussed and opportunities for future research have been identified in the context of size-effect.

FUNDAMENTALS OF MICROWAVE-MATERIAL INTERACTION

Microwaves with frequency 2.45 GHz are used in most of the material processing applications. Heating phenomena in microwave processing is entirely different from conventional processing. Heat generation with heat transfer from (inside) core of the material to (outside) surface of material takes place in microwave processing which is reverse in case of conventional heating [101–104]. Heat generation inside material during microwave processing reduces the effect of heat transfer and heating becomes more uniform and rapid [109]. The other added advantages of microwave processing are less time consumption, energy saving, eco-friendly processing and selective heating nature of microwaves. Microwaves interact with materials through mutually perpendicular electric field (E-field) and magnetic field (H-field) components of the electromagnetic radiation [109–112]. Thus, material heating during these interactions depends upon dielectric and magnetic properties of materials. Dielectric polarization and conduction losses are responsible heat loss mechanisms in nonmagnetic materials [6, 19, 101], while hysteresis loss, eddy current loss and residual losses are more prominent heat loss mechanisms in magnetic materials [6, 101, 113]. It is very difficult to predict contribution of each loss mechanism during microwave processing, however; combined effect of these mechanisms lead to volumetric heating of the materials [19, 48, 112]. The absorbed microwave power inside material is a function of dielectric and magnetic properties of materials. The microwave power equation as $P = 2\pi f \varepsilon_0 \varepsilon'' |\mathbf{E}|^2 + 2\pi f \mu_0 \mu'' |\mathbf{H}|^2$,where E is magnitude of the internal electric field, H is magnitude of the internal magnetic field, f is microwave frequency, ε_o is permittivity of free space, μ_0 is magnetic permeability of free space [11, 101]. The electrical energy is converted in to heat energy based on the dielectric loss factor (ε") and magnetic loss factor (μ") of the materials when exposed to electromagnetic field [15, 101].

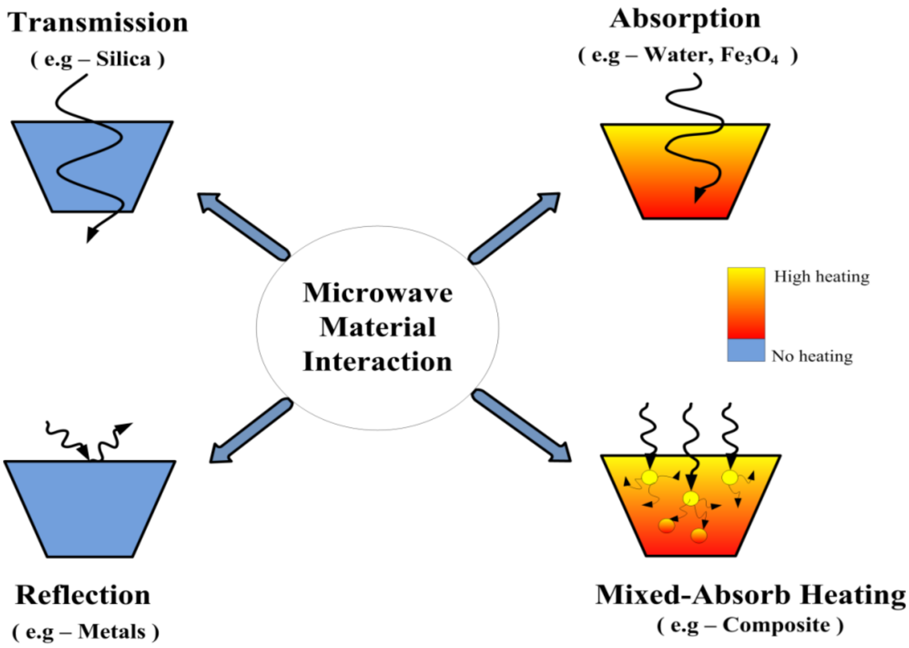

Figure 3. Principal groups of microwaves- materials interaction.

The other influencing parameter in microwave processing of non-metals/metals is the penetration/skin depth. The penetration/skin depth is defined as the distance into the material at which the incident field intensity reduces to $(1/e)^{th}$ (36.8%) of the surface value [37]. Skin depth ($\delta = 1/(\pi f \mu \sigma)$ where μ is magnetic permeability and σ is electrical conductivity) of bulk metals at room temperature is very small owing to their high conductivity; however, high effective surface area in metal powders improves microwave absorption at room temperature [5, 6, 37]. The microwave-material interactions have been categorized in four different principal groups [55, 101, 104, 110] as illustrated in Figure 3. The materials (like- metals) in which microwaves are not able to penetrate and get reflected are reflector to microwaves. Microwaves pass through materials having low dielectric loss factor (like- Teflon) without energy loss. Microwaves on the other hand, get absorbed in high dielectric loss factor materials, known as absorbers. The materials (like- composites) which have mixed phases of high and low dielectric loss factor materials absorbs energy selectively in high dielectric loss factor phase while other phase gets conventionally heated [55, 101, 104].

PROCESS PARAMETERS IN MICROWAVE PROCESSING

The control over microwave-material interaction during electromagnetic irradiation is mostly unexplored due to limited flexibility in available microwave applicators, instrumentations and understanding of physical phenomena involved [3]. The fixed sizes of microwave applicators and limited flexibility in levels of power, frequency and uneven distribution of microwave field intensity add complexity to process control [3]. These inherent challenges of processing materials using microwave have attracted researches over the year. The input parameters that can be controlled during microwave irradiation during material processing and their expected output responses are illustrated in Figure 4.

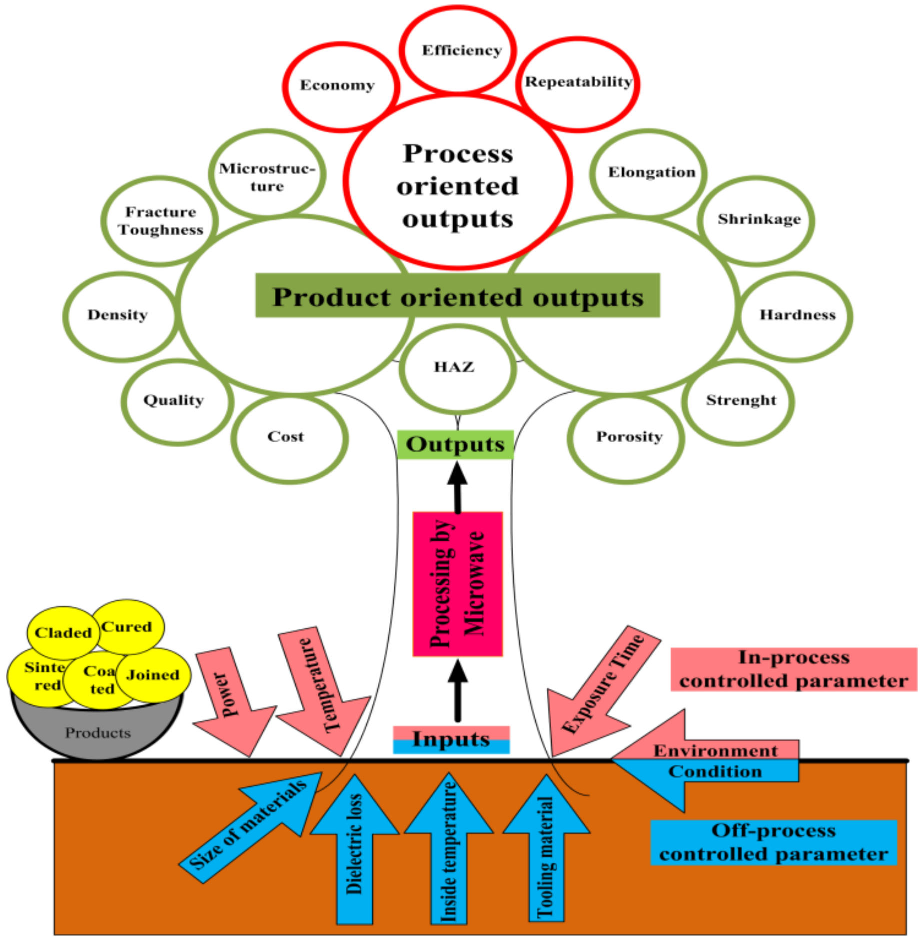

Figure 4. Parameters involved in microwave- materials interaction.

The input parameters in microwave material processing can be divided into two groups on the basis of their control during irradiation process:

(a) *In-process controlled parameters:* the input parameters, which can be controlled during the irradiation process, for example – exposure time, power etc.
(b) *Off-process controlled parameters:* the input parameters whose control is possible only before start of irradiation process, for example – material size, tooling materials etc.

Similarly, the output responses can be divided in two different groups as:

(a) *Product oriented outputs:* the output responses which can be controlled to improve product quality by controlling the input parameters, for example – mechanical and physical properties etc.
(b) *Process oriented outputs:* the output responses which can be controlled to improve the process by controlling the input parameters, for example – efficiency, economy, repeatability etc.

The desired output can be achieved during microwave material processing by proper selection of input parameters. The control on input parameter is possible only by selection of size of materials and tooling materials, tooling design, customized applicator and control of atmosphere inside the applicator. Among all these input parameters, size of material is easy to control prior to start of process and more significant than others as energy absorption into the target material and time of exposure greatly depends on it.

EFFECT OF MATERIALS SIZE ON MICROWAVE ABSORPTION

Generally, microwave absorbing engineering materials at frequencies above 1 GHz, are categorized in three major groups [29] (i) dielectric (high ε", for example- water, graphite, metal powders, metal oxides, multiwall nano carbon tubes, semiconductors, Sic fibers etc. [7, 87–93, 113, 114] (ii) magnetic (high μ", for example- ferrites compositions, carbonyl Fe and Ni powders [114, 115] and (iii) ferroelectric-seignettomagnetic (a relatively new family of materials combining ferro-, antiferro-, or ferrielectric properties with ferro-, antiferro-, or ferrimagnetic properties in a certain temperature range: high ε" and μ", for example- ferrite or carbonyl iron and lead zirconate titanate (PZT) [114]). Selection of tooling materials while processing engineering materials depend on their ε" and μ" as microwave power absorption is directly proportional to these properties. However, power absorption varies rapidly at higher temperatures due to temperature dependencies of ε" and μ" [114]. Different microwave processes reported so far for processing of engineering materials (metals, polymers, ceramics and their composites) are illustrated in Figure 5.

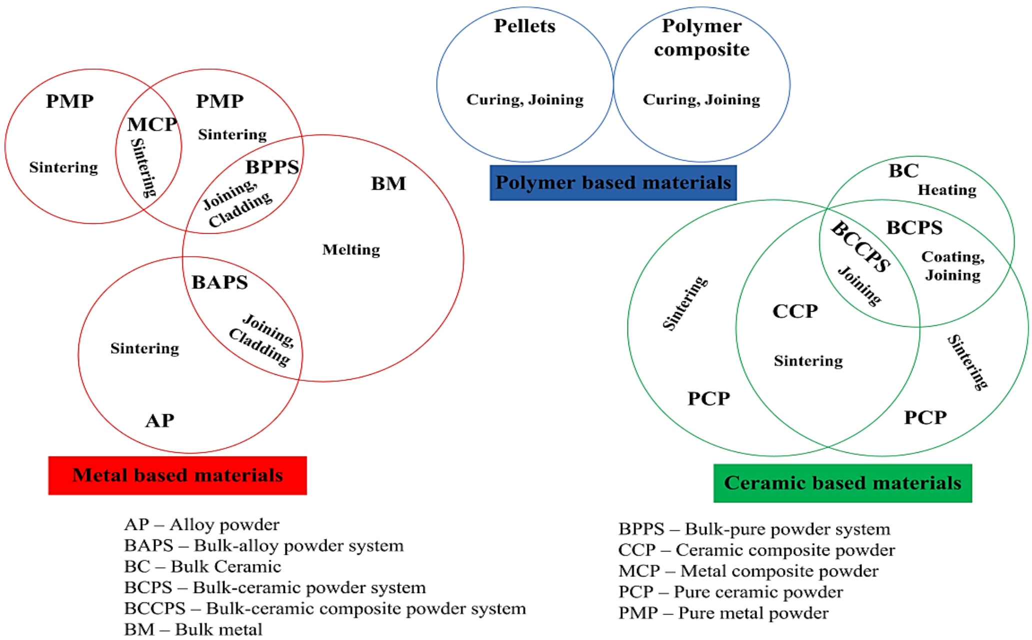

Figure 5. Materials and processes carried out by microwave energy.

It can be observed that different microwave processes mostly involve a combination of two same or different materials of different sizes (like- bulk-powder or powder-powder or fiber-resin or fiber-powder-resin). The effects of different sizes of constituents and decreasing sizes of engineering materials on microwave power absorption in different microwave processes have been discussed in this section.

Metals and Metal based Composites

Initially up to the year 1999, it was assumed that metal based materials are incompatible to microwave radiations as they reflect microwaves owing to their high conductivity and poor skin depth. The first interaction of microwave with metallic powders was reported during processing of ceramics by adding electrically conducting metal powders [116]. However, full sintering of metal powder compact was reported in the year 1999 [15]. Although, joining of metal based materials with satisfactory mechanical and metallurgical properties were reported by many authors [117–119], processing of bulk metals using microwave radiations was reported rarely. The year 2009 onwards, a few authors have reported microwave processing of bulk metals in the form of joining [120] and cladding [121]. Different constituents and sizes of metal based materials are used in sintering, joining and cladding using microwave radiations. Few selected results of particle size effect in microwave processing of metal powder are shown in Table1. Most of the metal powders are processed using microwaves in its pure the form or alloy or composite (mixture of metal and ceramic). The microwave power absorption in metal powders is affected by their magnetic or non-magnetic properties. The magnetic metal powders are more affected by magnetic field component of the incident microwave radiation, whereas, electric field component influences the non-magnetic metal powders more effectively [6, 19]. In case of non-magnetic metal powders (like- copper, aluminium etc.), the particle size largely affects the effective surface area of the green compact [5, 6]. The microwave power absorption in a green compact increases as the particle size decreases with the increase in effective surface area [6]. However, skin depth in green compact of smaller particles decreases due to reduced porosity which increases conductivity of green compact [5, 6, 9, 11, 26, 56]. It was also reported that the concept of microwave power absorption in a green compact can be well explained by the ratio of particle radius to skin depth (r/δ) [6]. Figure 6 illustrates the effect of radius of powder particle to skin depth (r/δ) ratio on microwave absorption in non-magnetic materials. There is an optimum value of ratio (r/δ = 2.4) at which the power absorption is maximum [6]. As the ratio (r/δ) decreases the green compact sintering approaches to uniform heating and heating rate increases. However, increase in ratio (r/δ) indicates non-uniform heating with decreased heating rate. In case of magnetic materials (like- iron, cobalt, nickel etc.), the effect of magnetic field has dominant role in heating of green compact, generally, up to particle size of 1µm [52]. As the particle size reduces below 1 µm, the magnetic field contribution in green compact heating gets reduced owing to decrease in size of magnetic domains of magnetic metal particles. Consequently, the heating of magnetic metal particles below 1 µm particle size becomes similar to non-magnetic metal particles [52]. The metal alloy powder heating/sintering behavior is similar to as discussed in the case of pure metals; however, sintering of metal matrix composites using microwave radiations is different due to presence of high dielectric loss factor reinforcements. Pure microwave power absorption in composites depends upon size of reinforcement as well as the size of metal powders. Few selected outcomes of nano reinforcements in MMC's are presented in Table 2.

Table 1. Few findings on particle size-effect in MW processing of metals and alloys.

Process	S/FM	MWP (W) @ *f*	FM size		Attributes	Outcomes		Reported by
			µm	nm		µm	nm	
MWS	Cu	500-1600 @ 2.45 GHz	6, 12, 18, 63, 383	-	SD (%)	95.4, 90.4, 90, 89.8, 90		[5]
					DP	0.88, 0.54, 0.48, 0.43, 0.39		
					MWHR	Improved with reduction in metal powder size		
	Al	166 @ 3.3 GHz	3.3	120	CL	5.76×10^{-10}	3.15×10^{-14}	[6]
					MWH	Reduced with reduction in PS		
	Ni	-	45, 63, 150	-	MWH	Improved with reduction in PS		[9]
	Sn	1100 @ 2.45 GHz	4,30,69	-	MWH	Faster with reduction in PS		[11]
	Cu		5,32,57	-	MWH	Faster with reduction in PS		
	W		2,6,11	-	MWH	Faster with reduction in PS		
	SS316L	1300 @ 2.45 GHz	56^a,45^b	-	UTS (MPa)	293^a, 296^b	-	[56]
					P (%)	4.4^a, 3.6^b	-	
					E (%)	9.5^a, 12.9^b	-	
MWJ	Cu plate/ Cu powder	900 @ 2.45 GHz	5^a, 40^b	-	UTS (MPa)	164.4^a, 135^b	-	[62, 109]
					MH (Hv)	78 ± 7^a, 72.9^b	-	
					E (%)	29.21^a, -	-	
					P (%)	1.92^a, 1.27^b	-	
	SS-316 plate / Ni powder	900 @ 2.45 GHz	5^a, 40^b	-	UTS (MPa)	338^a, 309^b	-	[61, 109]
					MH (Hv)	282^a, 290 ± 14^b	-	
					E (%)	-, 11.50^b	-	
					P (%)	0.85^a, 0.78^b	-	
	SS-316 and MS plate / Ni powder	900 @ 2.45 GHz	5^a, 40^b	-	UTS (MPa)	340^a, 324^b	-	[69, 109]
					MH (Hv)	170^a, 133^b	-	
					E (%)	-, 13.58^b	-	
					P (%)	0.80^a, 0.58^b	-	
	Inconel-625 / EWAC powder	600@ 2.45 GHz	50^a, 75^b	-	UTS (MPa)	347^a ,318^b	-	[71]
					FS (MPa)	615^a ,516^b	-	
MWC			40-45		Time(s)	600	504	[80–82]

AISI 304 SS / WC-12Co powders	1440 @ 2.45 GHz	100-200	Power	1.4 KW	1.1 KW
			MH(Hv)	1138±90	1563±53
			SWR	Improved with nano powder	
			FS	Improved with nano powder	

Abbreviations: MWS- microwave sintering, MWJ- microwave joining, MWC- microwave cladding, MWP-microwave power, S/FM –substrate/ filler material, Cu-copper, Sn-tin, W-tungsten, Ni- nickel, Mg- magnesium, Al- aluminium, SS- stainless steel, MS- mild steel, PS- powder size, MWHR-microwave heating rate , MWHA-microwave heating, CL- Conductive losses in (W·(V/m)−2), SD–sintered density, DP-densification parameter, UTS–ultimate tensile stress, YS–0.2%yield stress, MH–microhardness, E- Elongation , P- Porosity , SWR – sliding wear resistance, FS – flexural strength. Unclear or unavailable data are shown as'–'.

As the size of reinforcements decreases, microwave absorption in MMCs increases due to increase in effective surface area of reinforcements. The smaller size reinforcements act as a local susceptors and take part in selective microwave hybrid heating which provides more uniform heating in MMCs during microwave processing [17–22]. The rapid heating of green compacts of Cu powder (6-383 µm) [5], W powder (3-12 µm) [37] and Ni powder (45- 150 µm) [11] with finer particles were reported during microwave sintering in a multimode microwave applicator at 2.45 GHz. The size of copper particles and packing density were more responsible for heating of Cu compact while sintering of Cu with percentage porosity from 24 to 44% [5]. Higher heating rate of Cu compact was obtained at 6 µm and 44% porosity than 16 µm copper compact. The Cu and Ni compacts having lesser porosity with small particle size behave like a bulk piece and microwave absorption gets reduced drastically exhibiting a slow heating rate [5, 11]. Drastic reduction in sintering temperature of nano-tungsten powders was also reported during sintering of micro and nano tungsten powders [37]. Nano-sized particles got coupled rapidly with microwave energy and maximum sintering temperature could be achieved during microwave sintering which was however, less in comparison to conventional sintering. Consequently, processing time in microwave sintering was reduced by 90% than conventional sintering. The hardness of microwave processed compact was higher due to less coarsening during grain growth [11, 37].

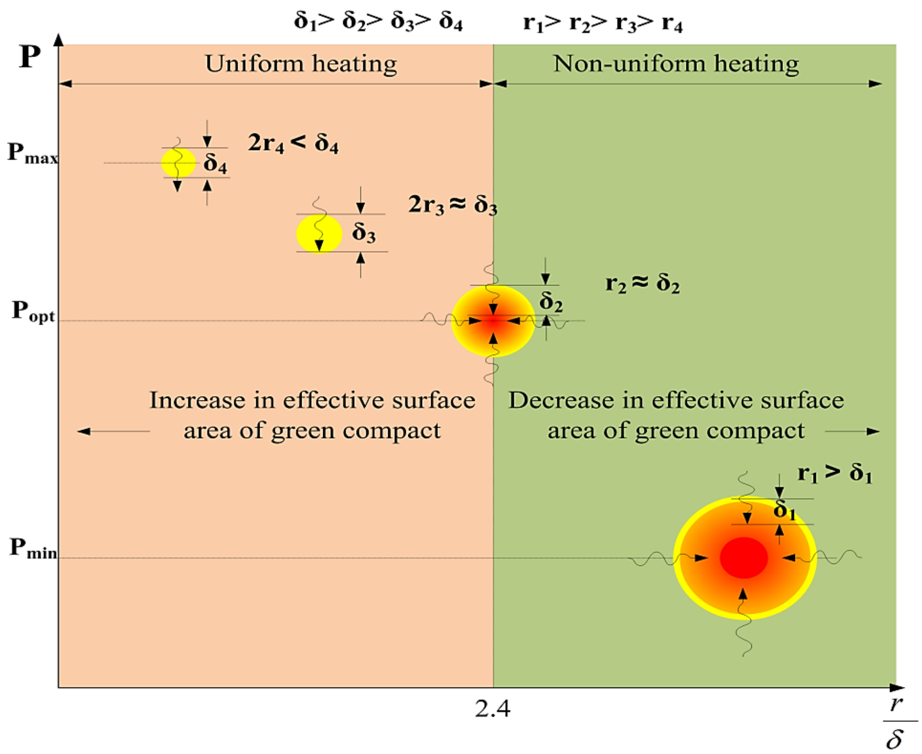

Figure 6. Microwave heating of non-magnetic metal.

Microwave sintering of copper compact having particle size from 0.7 to 22 µm and green density of 76% was reported in separate E and H fields at 400W [48]. The results revealed that an increase in particle diameter up to 6-8µm, causes the heating rate also to increase; however, further increase in particle diameter, decreases heating rate. Microwave absorption by individual particle (directly proportional to microwave absorption) and number of particles in compact (inversely proportional to microwave absorption) were the main influencing parameters to microwave absorption [48]. The Cu particles (diameter >2 mm) were non-uniformly heated in magnetic field; however, large electric field leads to more uniform sintering. Conversely, same size iron particles were heated more uniformly in presence of a magnetic field [48] and increase in particle size of ferro-magnetic metals increases the microwave absorption with rapid heating [11].

Table 2. Few selected outcomes of nano reinforcements in MWS of MMC's.

Composite	Power (W) @ *f*	Constituents size		Attributes	Outcomes		Reported by
		M-µm	RF - nm		Matrix	Composite	
(Mg/SiC)[a] (Mg/Al_2O_3)[b] (Mg/Cu)[c] (M–Mg)	900 @ 2.45 GHz	Mg- 60-300	Nano-SiC, Al_2O_3, Cu	Density (g/cm^3) P (%)	1.737 ± 0.001 0.17	1.753 ± 0.007[a], 1.748 ± 0.010[b], 1.809 ± 0.007[c] 0.11[a], 0.83[b], 0.13[c]	[23]
				MH(Hv)	40 ± 1	43 ± 2[a], 60 ± 4[b], 60 ± 3[c]	
				YS(MPa)	121 ± 2	157 ± 22[a], 154 ± 5[b], 194 ± 11[c]	
				UTS(MPa)	176 ± 2	203 ± 22[a], 213 ± 12[b], 221 ± 17[c]	
				FS(%)	5.4 ± 0.7	7.6 ± 1.5[a], 6.3 ± 0.4[b], 2.9 ± 0.4[c]	
Mg/ Al_2O_3 (M–Mg)	900 @ 2.45 GHz	Mg- 60-300	50, 300	Density (g/cm3)	1.738 ± 0.001	1.831 ± 0.005	[25]
				P (%)	0.07	1.04	
				MH(Hv)	47.0 ±1.3	73.7 ±1.1	
				EM(GPa)	45.0	54.4	
				YS(MPa)	116 ± 11.1	157±20.3	
				UTS(MPa)	168 ± 10	211±21	
				Ductility (%)	9.0 ± 0.3	3.0±0.3	
Mg/Fe_2O_3 (M–Mg)	900 @ 2.45 GHz	55	50	Density	Improved with nano Fe_2O_3 RF		[31]
				MH (Hv)	Improved with nano Fe_2O_3 RF		
Al–ZrB_2 (M–Al)	900 @ 2.45 GHz	-	<12	MH(Hv)	-	63	[32]
				UCS (MPa)	-	270	
Al/ Si_3N_4-3% (M–Al)	-	Al - 10	Si_3N_4 - 10-30	MH(Hv)	38 ± 3	77 ± 2	[33]
				YS(MPa)	70 ± 4	127 ± 4	
				UCS(MPa)	305 ± 3	364 ± 2	
(Mg-Al)/BN	-	Mg- 60-300	BN-50	UTS(MPa)	Increases with addition of nano RN		[30]
				UCS(MPa)	Increases with addition of nano RN		
				MH(Hv)	Increases with addition of nano RN		

Abbreviations: MWS-microwave sintering, RF-Reinforcements, UTS–ultimate tensile stress, YS–0.2%yield stress, MH–microhardness, P- Porosity , UCS–ultimate compressive strength, EM–elastic modulus. Unclear or unavailable data are shown as'–'

In case of aluminium, formation of Al_2O_3 affects the power absorption in green compacts [6]. Decrease in particle size of Al, reduces the microwave heating depending upon the purity level of Al. Nano size Al particles favors formation of more Al_2O_3 than micron size particle based on purity level mentioned (submicron – 81% and micron- 99.8%). The observed oxide layer thickness was higher in nano size particles [6]. The higher oxide shell thickness reduces microwave heating and micro-Al particles were heated more rapidly than the nano or submicron particles [6, 8]. Sintering of Ni (45 µm), 316L stainless steel (45 µm), Co (5 µm), Cu (75 µm), and Fe (45 µm) were reported using microwave and conventional sintering processes. The shrinkage parameter for microwave sintering was higher than conventional sintering and this effect is predominantly noticeable in Co powder due to smaller particulate size [52]. The in-process control of particle size was explored by increase in the amount of dopants (like- HfO_2 and Y_2O_3) which increase the surface area and decreases the particle size of tungsten powder from 350 nm to 80-100 nm with addition of 1.14 wt. % of HfO_2 [53]. Size-effect in microwave sintering of metal alloy powders was reported by very few authors. Microwave sintering of 316L stainless steel powders (particle diameter- 56 µm and 45 µm) were reported in 90% Ar/10% H_2 atmosphere for 1 hour at 1300 W by Ertugrul et al. [56]. The sintering results revealed that densification parameters (increased by 29.41%), yield strength (increased 1%), shrinkage (increased 1.1%) and porosity (reduced by 0.8 %) were improved in the compacts of particle size 45 µm than particle size 56 µm [56]. The 2712 alloy (Al -3.8Cu -1Mg -0.8Si -0.3Sn) powder having an average particle size of 105 µm was found coupled well with microwave during sintering process and resulted in rapid heating rates with a 55% decrease in process time [16]. Microwave sintering results of aluminium MMCs (matrix- Al, 7-15 µm; reinforcement- Ti, 20 µm; and filler- SiC, 50 nm) were reported with improved mechanical and metallurgical properties [17]. The addition of nano size SiC particles in hybrid composite of Al/(Ti + SiC) resulted in reduction of porosity level and improved yield stress of the matrix [17]. Poor properties in copper composite with graphite (30% by vol., 50 µm) were reported due to graphite particle agglomeration which reduces the microwave absorption capacity and heating rate [20]. Copper (12 µm) –TiC (50 µm) –graphite (11 µm) hybrid composites were reported with lower relative density as the percentage volume of graphite increases from 5% to 10%. The increase in porosity was attributed to non-uniform distribution and agglomeration of coarse size graphite particles [18, 21]. The nano size Cu particles (50 nm) improve density and hardness of Mg/Cu composite by filling the voids in Mg (60–300 µm) matrix and good interfacial integrity between matrix and nano-size Cu particles [22]. However, increase in percentage volume of nano-size Cu particles in composite resulted in higher tendency of agglomeration. The nano size reinforcement like SiC, Cu, Al_2O_3 [23] and Y_2O_3,Ni [24] in magnesium matrix by up to 1% volume; improve hardness, ultimate tensile strength, 0.2% yield strength and ductility compared to bulk magnesium. Further, improvement in mechanical properties of magnesium nano composites was reported compared to bulk magnesium by adding sub micron size Al_2O_3 (4.0% vol.) and nano size Al_2O_3 (1.0% vol.) [25]. The presence of sub micron size Al_2O_3 (4.5% vol.) affects the ductility of composite which was overcome by increasing the addition of nano size Al_2O_3 and reducing the sub micron size Al_2O_3 [25]. Microwave sintering of magnesium MMCs (matrix- Mg-55 µm; reinforcement- Fe_2O_3-50 nm) was carried out in industrial microwave oven at 900 W. The authors reported that the addition of nano size reinforment improved the relative density and micro hardness. The improvement in the hardness of nano composites attributed to the resistance offered by the Fe_2O_3 nano particle against indentation [31]. The significant improvement in microhardness and ultimate compressive strength were observed in Al–ZrB_2 MMCs by addition of nano size (<12nm) ZrB_2-10% reinforcement [32]. The addition of nano size reinforcement in MMCs significantly increases the mechanical and metallurgical properties of the composites attributed to the uniform heating rate and improvement in the sintered density; However the percentage of addition of reinforcement mostly governing the properties of the composites [31–33].

The effect of clad powder size was also reported in microwave cladding of austenitic stainless steel by using micro [78] and nano size [80–82] WC-12Co powder particles in a multimode industrial applicator at 2.45 GHz. The decrease in WC-12Co powder size (from micro to nano level) resulted in improved hardness and wear resistance of the clad surface. This was attributed to enhanced uniform distribution and effective surface area of nano size clad powder for better microwave absorption. Uniform plastic deformation and material removal in small fragments with smooth surfaces were reported with nano size powder clads than micro size powder clads [78–82]. The effect of specimen size during joining of Inconel-625 alloy was reported in terms of exposure time [70]. The exposure time for small size specimen (20 × 6 × 3 mm) was 9 minutes, whereas large size specimen (102 × 12× 6 mm) was joined in 21 minutes. The effect of size of filler material was reported by researcher in microwave joining of Inconel-625 alloy. The use of small size filler material (50µ) at 600 W power levels resulting in increasing in the ultimate tensile strength (347MPa) and flexural strength (615 MPa) compare to large size filler material (75µ). The use of 75µ filler material shows ultimate tensile strength and flexural strength are 318 MPa and (516 MPa).The increase in the properties attributed to the better heating rate is achieved due to use of reduced filler size [71]. The effect of particle size (macro, micro and nano) of filler powders of SS-316 and Ni based powders were reported during joining of SS-316, mild steel and their combinations using 900W microwave irradiations at 2.45 GHz [67–71]. The experimental results revealed that homogenous and dense joint through metallurgical bonding with the substrate were developed by using nano fillers. The mechanical and metallurgical properties of joints were reportedly improved by use of nano powders than other powders. In another experimental study, melting and flow of SS-316 micro powders were reported during butt joint of SS-316 and MS bulk metal pieces [122]. However, better joints were developed by control over size of filler materials, exposure time and irradiated microwave power.

Ceramics and Ceramic based Composites

Generally, ceramic materials interact with microwave radiations at room temperature differently. Most of the ceramics (like- SiC etc.) are good absorber of microwaves depending upon their dielectric loss factor; however, some of ceramics (like- Al_2O_3, MgO etc.) are transparent to microwave radiations [36, 38, 39, 45, 105, 111]. The transparent ceramics start microwave absorption above a certain temperature which is known as critical temperature of ceramic [105, 111]. The electric filed component of microwave induces dipolar losses and conduction losses in ceramics. These losses include active heat loss against friction, inertial and molecular forces due to dipolar orientation and electron movement in ceramic materials, these losses are considered prime causes of ceramic material heating in microwave field [105, 111]. At elevated temperatures, ceramic materials absorb microwaves more rapidly with higher heating rate. This results in thermal runway and ultimately leads to cracking of ceramics due to induced non-uniform thermal stresses [111]. It was reported by several authors that the use of micro and nano size ceramic powders during microwave sintering improves heating uniformity and reduces chances of damages [38–42]. Few selected results of ceramics and ceramic composites are shown in Table 3. In CMCs, two different dielectric loss factor ceramic powders get processed. Mostly, reinforcements which are added in CMC, possess high dielectric loss factor and control heating and bonding between reinforcement and the matrix. These reinforcements heat the matrix with selective hybrid heating [45–47, 49–51].

Table 3. Few findings on effect of particle size during microwave processing of ceramics and ceramic composites.

Process	S	PS		Property	Outcomes		Reported by
		µm	nm		µm	nm	
MWS	HA	5.01 ± 1.02	168 ±0.086	CS (MPa)	88 ± 29	395 ± 42	[28]
				MH (GPa)	3.8 ± 0.42	8.4 ± 0.4	
				FT (MPa $m^{1/2}$)	0.8 ± 0.07	1.9 ± 0.2	
				SE(mJ/m^2)	71.92 ± 1.82	97.30 ± 4.28	
	ZrO_2/ Al_2O_3	-	35 /380	FT (MPa $m^{1/2}$)	Increases with decrease in PS		[49]
				Density	Increases with decrease in PS		
				MH	Increases with decrease in PS		
	WC/ CO-7.5%	1-3^a	500^b,100^c	Density (g/cm^3)	lower	15.1^c	[47]
				MH (Hv)	lower	1800^b	
				FT (MPa $m^{1/2}$)	lower	15^b	
	Al_2O_3 /SiC -3%	-	SiC- 40^a, 100^b, 500^c	Relative density (%)	-	93.4^a. 98.4^b, 97.4^c	[51]
				MH (GPa)	-	14.53 ± 0.34^a, 18.40 ± 0.24^b , 17.63 ± 0.75^c	
				FT (MPa·m1/2)	-	5.29 ± 0.32^a, 4.97 ± 0.30^b, 5.31 ± 0.32^c	
	SiC/epoxy	66.7±14.47	71±34	MA & CD	Improved with reduction in SiC PS		[45]
	BaTiO3	1.33, 0.19, 0.066	-	RP	Increases with decrease in PS		[46]
				DL	Decreases with decrease in PS		
MWH	Al_2O_3	0.0315,1.5,2.4,3.15 in mm		MWH	Increases with decrease in PS		[4]
	Fe_3O_4	0.0815,0.75,1.5 in mm		MWH	Decreases with decrease in PS due to conversion of Fe_3O_4 in non-magnetic particle (Fe_2O_3)		

Abbreviations: MWS- microwave sintering, S-substrate, PS- particle size, HA- hydroxyapatite, CS-compressive strength, MH-microhardness, FT-fracture toughness, SE-surface energy, MWH- microwave heating NH-nanohardness,YM- Young's modulus, MA-Microwave absorption, CD- complex dielectric, RP-relative permittivity, DL- dielectric loss', Unclear or unavailable data are shown as'–'

The effects of size of Al_2O_3 and Fe_3O_4 ceramic powders during microwave heating were reported by Standish et al.[4]. The small particles Al_2O_3 (0.0315 mm) absorb more microwave energy with higher heating rate than coarse particles Al_2O_3 (3.15mm). On the other hand, coarse Fe_3O_4 (1.5mm) particles absorbed microwave more rapidly than fine particles (0.0815 mm) during microwave exposure. This reverse phenomena of size effect was attributed to conversion of Fe_3O_4 (high ε'') into Fe_2O_3 (low ε'') due to oxidation. The small particles rapidly get converted due to oxidation than larger particles of Fe_3O_4, thus, coarser particles get heated more rapidly than smaller particles [4, 10, 12]. A negligible change in heating rate of Al_2O_3- Fe_3O_4 mixtures were reported due to variation in Fe_3O_4 particle sizes. Microwave sintering of Hydroxyapatite (HA) compacts were reported for dental applications [34, 35]. The effect of grain size (0.168±0.086, 0.52 ± 0.092, 1.16 ± 0.17, 1.48 ± 0.627 and 5.01±1.02 µm) variation was evaluated for mechanical properties of HA compacts. The increase in gain size resulted in requirement of higher temperature and time to sinter HA compact. The mechanical properties like compressive strength, microwave hardness and Indentation fracture toughness were reduced 73.22%, 52.27% and 58.57%, respectively, as the grains size was increased. The joining of Al_2O_3–ZrO_2 composite blocks (15×4×4, mm3) were reported in form of butt joint using sodium silicate glass powder which were used as a sealing material. The reduction in hardness of joint interface was attributed to larger interface thickness of sample and larger powder size used. Further, decrease in particle size resulted in increased hardness of the joint interface [72]. The ceramic matrix composites of SiC powder having particle size from 66.7 ±14.47µm to 176±201 nm using metal powder of Cr/Mn/Al/Co/Zn/Ni/Ti (10%) as reinforcements and epoxy (20%) as binder were reported with improved microwave absorption properties as particle size of SiC decreases from micro to nano level [45]. The particle size of magnetic materials is directly proportional to the number of magnetic domains present in it. The increase in particle size, increases the tuning difficulties due to complex domain states of microwave properties of polycrystalline ceramic ferroelectric ($BaTiO_3$) [46]. Consequently, smaller particles (0.26 µm) of ceramic $BaTiO_3$ were reported with lower loss tangent and increase in relaxation frequency than larger particle (14.4 µm). Similar result was also reported for $BaTiO_3$ powder-polymer matrix composites for average particle size of 1.33 µm, 0.19 µm, and 66 nm [46]. The microwave sintering of WC-Co exhibited improvement in the microhardness and fracture toughness using submicron (500 nm) particle size. The authors reported that use of nano size (100 nm) Co particles improved density of the compact but slightly deteriorate the hardness and fracture toughness due to grain growth during microwave heating [47]. Similar results have been observed during microwave sintering of Al_2O_3 /SiC -3% composites. The addition of nano size (100 nm) SiC reinforcement shows improvement in relative density and microhardness compare to 40 nm and 500 nm. It was observed that the SiC with 40 nm particle exhibits micro pores resulting in poor density and hardness whereas SiC with 500 nm shows coarser grain with inhomogeneous microstructure [51].

Polymers and Polymer based Composites

Microwave heating of polymer based materials are quite difficult as most of the polymers are transparent to microwave energy at room temperature [104, 110]. The microwave absorption in polymers depends upon changes in their chemical structures during the processing. In case of thermosets, microwaves abortion stops as soon as the cross linking in internal structure of thermosets takes place. On the other hand, thermoplastics behave as transparent depending upon their degree of cyrstallinity [104]. Size of polymer affects the processing time during microwave irradiation; also presence of more polymers needs long processing time [87, 88, 93, 94]. Few selected results of microwave curing of polymer composites are shown in Table 4. Thicker pellets take more curing time and bulk polymer is difficult to heat by microwave energy compared to small size pellets. The processing time can be controlled by the addition of conductive fillers like magnetic particles, CNTs, carbon black, graphite nano-platelets (GNP), metal particles etc. The microwave absorption in PMCs are governed by high dielectric loss factor constituents like carbon fibers [89–100]. The use of primer helps to improve the microwave heating of polymer by enhancing bond strength [65]. However, in case of poor dielectric loss factor fibers (like –lead, aramid etc.), the heating will be controlled by the properties of the matrix [104].

Table 4. Few findings on effect of reinforcement particle size during microwave curing of polymer composites.

PMC constituents	RF particle size		Property	Outcomes		Reported by
	µm	nm		P	PMC	
PCL/nHA-20%	-	100	UTS(MPa)	1.83	4.5	[90]
			FS (MPa)	3.71	7.2	
			SH	49.91	75.47	
			IS (KJ/m^2)	2.25	0.89	
			ET (s)	483	368	
ER (EPON 862) / CNTs - 0.3 wt%	-	10–20	FS (MPa)	102	128	[91]
			EM (GPa)	1.96	2.32	
(HDPE+ 20 wt% CNTs)[a]	-	5–15	UTS (MPa)	-	19.7[a], 2.53[b]	[92]
(PP+ 20 wt% CNTs)[b]	-		EM GPa	X	295X[a], 787.8X[b]	
(PEEK)/ SrTiO3 powder -27 wt%	5[a]	<100[b]	DC (ε_r)	-	5.27[a], 5.9[b]	[98]
			LT -tan δ	-	0.0037[a], 0.0278[b]	
			TCDC($\tau_{\varepsilon}r$)	-	12[a], 444[b]	
			VH (kg/mm^2)	22	33.4[a], 37.1[b]	
polyester resin/NiCFs – 4 wt %/MWCNTs-1 wt%	NiCFs - 9	MWCNTs - 10	CP-ε	2.68	14.90 -16.80	[89]
CNF/ER Ni-coated CNTs/ER	-	CNFs-150 -	MA	Achieved better MW absorption by using nano carbon fibers		[105, 110, 111]

Abbriviation: P-polymer, PMC- polymer composite, PCL- polycaprolactone, nHA- nano Hydroxyapatite, ER- epoxy resin, CNTs-carbon nanotube,PR- polyester resin, PEEK-polyether ether ketone, HDPE- high-density polyethylene, PP- polypropylene, UTS-ultimate tensile strength,FS-flexural strength, SH-shore hardness, IS-impact strength, EM- elastic modulus, ET- exposure time, DC-dielectric constant, LT-loss tangent, TCDC- temperature coefficient of dielectric constant, CP-complex permittivity, VH-vickers hardness MA-microwave absorption, Unclear or unavailable data are shown as'–'

The polycaprolactone (PCL) composite foams was developed by incorporating nano Hydroxyapatite (100 nm) as reinforment. The authors have reported that addition of nano reinforcement decreases the processing time compare to neat polycaprolactone at 540 W microwave powers. The significant improvement in the ultimate tensile strength, flexural strength and shore hardness were reported by addition of nano Hydroxyapatite by 20% compare to neat PCL. This is attributed to the presence of nHA in composites restrict the mobility and deformability of the PCL. The presence of hard particle (nHA) is responsible for increase in the hardness value of the composite. However the presence of stiff nHA reduces the impact strength of the composite [90]. The researchers have reported that the addition of nano size reinforcement in the matrix increases the properties of the composite compare to neat polymer. The addition of nano size CNTs reduces the processing time as these are high dielectric materials which absorb microwave energy and heated very quickly. Volumetric and uniform heating of reinforcements and matrix resulting in improvement in the composite properties [91, 92]. The glass matrix composites were developed using borosilicate glass powder (20 µm) as a matrix and various metallic filers like Mo (2 µm), W (40 µm), Ti (40 µm), Ni (12.6 µm), Fe (40 µm) and Al (22.5 µm) as reinforcements by 10% volume. It was reported that small molybdenum powders agglomerated as compared to other metallic reinforcements [123]. The molybdenum glass matrix composite interfaces were appeared good without cracks, whereas in all other composites, micro pores were observed at the matrix-reinforcement interface [123, 124]. It was seen reported that increase in the thickness of CFRP laminates, the microwave absorption increases which reduced the processing time [87]. The size of fiber, volume percentage of fiber and its distribution control microwave absorption in PMCs. Thicker and high fiber content deteriorate composite quality as thick fibers are poor microwave absorber and results in poor adhesion. The use of nano and micro size fibers/fillers provide rapid absorption of microwave and excellent adhesion bonding between fiber and polymer [87–89, 93, 98–100]. Microwave absorption in polymer composite depends upon the amount of filer added. Improved mechanical properties of PMCs were reported with use of nano fibers due to higher aspect ratio and high specific surface area [89–92, 125, 126].

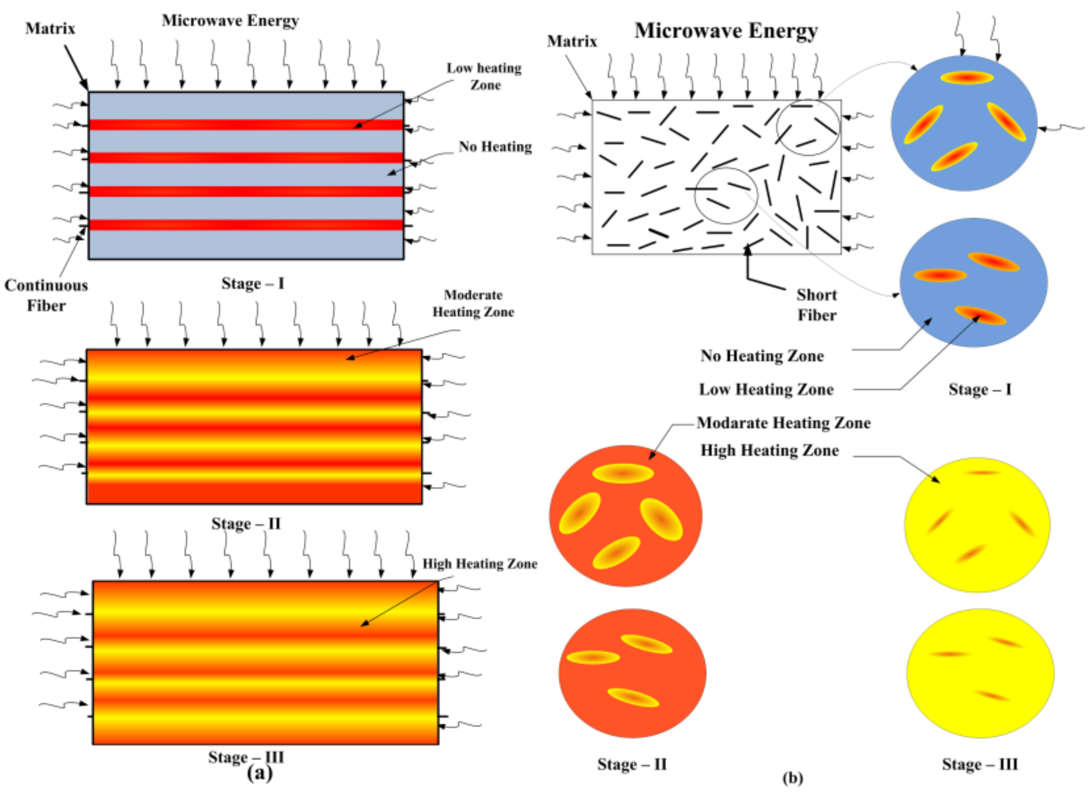

Figure 7. Microwave heating of polymer composite with (a) continuous fibers and (b) short fibers.

The percolation threshold (% volume of conductive filler at which polymer composite forms conductive channels through it) of a PMC is directly proportional to thickness while inversely proportional to diameter and aspect ratio of conducting fillers [127]. The addition of small size magnetic fillers in PMCs, improves microwave absorption due to formation of magnetic bridges by the magnetic particles. This enhances interaction with microwaves effectively [89–92, 128–133] and results in better heating. More hotspots inside a given volume improve heating effect and uniform heating of the bulk is observed. The nano size Ni fillers in NiCuZn ferrite/Ni/polymer PMC improves curing and mechanical properties of functional composites [99]. The effects of long conductive fibers and short conductive fibers on microwave curing have been illustrated in Figure 7. As the irradiation begins, initially, microwave energy is absorbed by conducting fibers (both short as well as long fibers) in PMCs (Figure 7. a &b stage-I). Further, heat is transferred to polymer matrix by conducting fibers and interfaces get cured (Figure 7. a&b stage-II). Microwave absorption in polymer matrix starts depending upon their critical temperature and degree of crystallinity. Microwave energy is more absorbed by short conducting fibers than long fibers due to high effective surface area (Figure 7. a &b stage-II). Thus, small size fibers provide better curing during microwave irradiation.

CHALLENGES AND OPPORTUNITIES IN METAL PROCESSING DUE TO SIZE- EFFECT

The dominance of the metal-based materials in engineering applications is yet to be overcome in spite of rapid developments in other new materials. However, processing of metal-based material systems by microwave energy is an inherent challenge to the scientific community. However, application of the size-effect concepts could open up new opportunities in this area too.

Table 5. Challenges during microwave processing of metal based materials.

Basis	Challenges in metal based materials processing
Agglomeration	Improper distribution of smaller size metal particles in metal based materials leads to agglomeration and non uniform heating
Dopants	Control of size and purity of metal particles during microwave processing by innovative methods like - addition of dopants
Filler materials	Use of optimum quantity of filler materials maintaining required porosity for better microwave absorption
Nano-size metal based data handbook	Unavailability of nano-size metal based materials microwave processing data hand book
Oxidation	Control of processing environment to reduce oxidation effect in metal based materials
Physics	Inadequate understanding of the physics of interaction phenomena of microwave-metal based material
Mathematical models	Rarely available mathematical models to simulate behaviour of microwave processing effects in metal based materials

The metal size (particle/bulk) affects microwave absorption in metal based materials due to their skin depth and high conductivity. Some of the processing challenges in microwave processing of metal based materials reported by various authors have been tabulated in Table 5. The reported data by various authors on size effect in microwave processing of metal based materials have revealed that deceasing size of metal based materials have improved the properties of developed products. The rapid response, eco-friendly nature and less time consuming behaviour of microwaves have attracted researchers to develop microwave processed products for industrial usages. Further, the better understanding of process physics, mathematical models of the process and control over environmental conditions in microwave applicator will help in achieving better quality, higher efficiency and robust process design. Some of the research opportunities of the process in metal based material processing due to size effect have been identified and indicated against different microwave processes as follows:

(a) Sintering
- In-process mixing of reinforcement and matrix
- Control of particle size during process
- Processing of MMCs with various additives like Al_2O_3, CNTs etc. with different sizes
- Processing of alloy metal powders to study the size effect
- Effect of powder size in mechanical and metallurgical properties of sintered compacts

(b) Joining
- Use of nano-size powders in joining of similar and dissimilar bulk metals.
- Effect of filler powder size of same or different metal powders in joining
- Effect of filler powder size in joining of different size of metallic substrates such as plates, pipes etc.
- Effect of filler powder size on mechanical and metallurgical properties of joints

(c) Cladding
- Effect of clad powder size in wear properties of metal based materials cladding
- Effect of clad powder size in mechanical and metallurgical properties of developed clads

(d) Heating of bulk metals
- Effect of bulk metal size in heating applications like melting, forming and casting
- Effect of bulk metal size in microstructure control during heat treatment processes

CONCLUSIONS

In microwave processing, the particle size of the substrate plays a significant effect on processing of materials and its properties. Decrease in particle size, enhanced metallurgical and mechanical properties of the materials were reported during microwave sintering, cladding, melting and curing. In microwave metal processing techniques such as joining and casting, size effect is rarely reported. Bulk ceramic, polymer, CMC's and PMC's materials processing using microwave also needs to be investigated for size-effect with further details. This provides an ample scope of research in the area of microwave processing and control over size of material for better product development. Some of the major conclusions have been drawn are as follows:

1. Reduction in particle size from micro to nano, the sintering temperature decreases drastically resulting in improvement in the bulk properties such as density, strength, hardness of the specimen in metal, ceramic and their composites compare to conventional thermal heating.
2. Use of small size powder particle in microwave cladding and joining processes shows significant improvement in clad surface and joint properties in metals.
3. The presence of high dielectric submicron size reinforcement in composites such as MMC's, CMC's and PMC's resulting in faster processing/curing time due to higher microwave absorption leading to uniform and volumetric heating of composite which improved propertied of the composites.
4. Handling of nano-size powder particle/fibers is difficult and non-uniformly distribution resulting in agglomeration leading to reduction in microwave absorptivity.
5. Optimization and controlling of process parameter is to be required before microwave processing of materials because it lead to over cured or under cured the materials which would certainly affect the mechanical and physical properties.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the help and guidance provided by Dr. Radharaman Mishra, IIT Roorkee and Dr. Apurbbakumar Sharma, Professor in Mechanical and Industrial Engineering Department, Indian Institute of Technology Roorkee, Uttarakhand, India in the manuscript.

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