

Comparative Investigation of Microwave Hybrid and Conventional Sintering on the Densification, Microstructure, and Phase Evolution of Magnesium-, Vermiculite-, and Tungsten-Based Powder Metallurgy Composites

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Abstract: The process of microwave hybrid sintering has come out as a potential substitute for the traditional method of sintering by means of furnace due to the advantages offered by rapid volumetric heating, energy efficiency and densification characteristics. In the present research work, a comparative study on the densification performance, microstructural analysis, surface integrity and phase formation of microwave sintered and conventional furnace sintered magnesium, vermiculite and tungsten powder metallurgy composites have been conducted. Compacts were prepared using powder metallurgy techniques and subjected to processing under similar conditions through microwave hybrid heating and conventional furnace sintering. Effect of sintering temperature (500-900°C), reinforcement content (1.0-2.0 wt.%) and holding time (3-6 minutes) on densification and porosity were studied systematically. The microstructural studies were conducted using optical microscopy and the phase formation was analyzed using X-ray diffraction (XRD). It has been observed from the experimentation that the process of microwave sintering resulted in better densification with bulk density of 7.88 g/cm³ and porosity of 20.42%. Optical microscopy showed improvements in particle bonding, pore refinement, better formation of necks, and homogeneity in the microstructure in comparison with the traditionally sintered samples. XRD results proved successful creation of crystalline phases in both composite systems, where the effect of reinforcement was very evident in the composition of phases and peak intensity. This superiority of microwave sintering can be explained by volumetric heating, which facilitates fast diffusion and homogenization of particles. Generally, microwave hybrid sintering shows great prospects for production of composites based on magnesium, vermiculite, and tungsten.

Keywords: Microwave and Conventional Heat Treatment; Vermiculite; Magnesium; Tungsten; X-ray diffraction analysis

1. Introduction

The wide adoption of advanced engineering materials across sectors like aerospace, automotive, nuclear power, and structural engineering has made this growing need for manufacturing techniques that are efficient, environmentally sustainable, and able to cut down processing time a lot. Traditional fabrication methods still struggle with the peculiar processing problems tied to these materials, mostly because their thermal behaviour is complex and it actually matters a great deal for the quality, and the overall performance, of the final products. Heat directly affects the grains of the materials and a temperature gradient exit across the material which changes the microstructure and mechanical properties [1]. A lot of manufacturing methods for advanced materials, kind of rely on heat being applied during processing. So, conventional thermal processing, mostly depends on how well heat can be moved from the energy source to the material thats being worked on. But the issues that show up with these heat-transfer approaches have pushed researchers toward other processing technologies, that can make advanced materials in a more effective



way, and often with better efficiency. Besides boosting overall process performance, these newer techniques are also expected to offer quicker production speeds. This matters because turnaround times keep getting more demanding all the time. The microwave material processing technique is one such solution which offers significant time saving and hence can be cost-effective and more importantly clean and environment-friendly [2].

Microwave technology was at first made mainly for communication. But then in 1946, Percy Spencer noticed that microwave energy could make materials heat up, and research into the thermal side kind of took off right after that, by the 1950s it was pretty much established and solid. Afterward the benefits of microwave heating were used, in a wide range of industrial tasks, like food cooking, thawing, and also tempering. People also tried it for wood curing, for medical waste treatment, for rubber tyre devulcanization, for sewage sludge processing, and even for regeneration of spent activated carbon. It was also used for handling petroleum industry residues, plus a number of nuclear related uses, and similar applications too [3]. Further, the microwave (MW) heating gained very fast popularity for processing of ceramics, polymers, metallic based materials and advanced materials such as metal matrix composite (MMC), ceramic matrix composite (CMC), and polymer matrix composite (PMC) [4].

The uniform heating characteristic of microwave yields better polymeric products than the conventionally cured. For example, thin as well as thick PMCs were processed with better mechanical properties and better bonding between fiber and matrix in thermoset and thermoplastic polymers has been reported using selective microwave heating [5]. In selective heating, only a selected volume of the target material is exposed to microwave radiation. This mode of heating was found more effective in high conductive fibers [6]. The addition of conducting particles and a coating on nanofibers within the carbon fiber hybrid composite really improved the bonding strength, mostly because the fiber phase now absorbed microwaves more efficiently, and it created better adhesion between the materials. The microwave fusion joining of PMCs was also reported by some researchers [7]. Very less literature is available in microwave processing of natural fiber PMC. The uniform microwave curing had improved flexural strength of natural fiber PMC. The microwave heating application was extended for joining of natural fiber composites which provides a feasible solution for better joint properties

Intensive research work has been reported in microwave processing of ceramics as they inculcate improved properties by coupling with microwave energy at room temperature; however, at elevated temperatures enhancement in diffusion rate has been observed with non-thermal effects [6]. In addition to plain thermal heating, microwaves are also able to cause non-thermal, or microwave-specific, effects when strong electromagnetic fields interact with ceramic materials in a more direct way. Kind of similar observations have been noted during polymer processing as well as in certain chemical reactions, though the exact mechanisms are still not really clear enough. A few studies have shown that microwave irradiation can substantially change reaction rates even in situations where no obvious physical changes can be seen. Also, microwave-assisted fabrication of ceramic matrix composites, CMCs, has been demonstrated to improve material properties, which suggests this technique may be useful for advanced composite manufacturing. Processing of nano-CMCs with nano reinforcements were also reported with enhanced densification rate, reduced porosity, improved sinterability and better mechanical properties [8].

Heating mechanisms in microwave materials processing

Heat generation during the interaction between microwaves and materials involves multiple physical processes. Exposure to microwave energy causes the electric and magnetic field components to influence the behaviour of dipoles, free electrons, magnetic domain walls, and electron spins by altering their orientation, movement, or position within the material. Depending on the material properties, one or more of these mechanisms may occur simultaneously during microwave processing. The following section briefly explains these heating mechanisms with reference to a representative elemental volume of the material.

Heating mechanisms in non-magnetic materials

Non-magnetic materials tend to couple mostly with the electric part of microwave energy not really with the magnetic one. For this group of materials, the microwave warming mostly comes from two routes, sort of conduction

losses and dipolar losses. Conduction losses show up as the main effect in metals and other super conductive materials, meanwhile dipolar losses take over in insulating dielectrics, such as ceramics, polymers, water, and several other nonconductive substances.

Dipolar Loss

Dipolar loss is basically the main heating route in dielectric materials that have polar molecules, meaning those molecules can respond when an external electric field shows up. Think water, ceramics, polymer matrix composites (PMCs), ceramic matrix composites (CMCs), and also many kinds of food materials. When these are exposed to an alternating microwave electric field, the molecular dipoles keep rotating, trying to track and align with the field that keeps changing. In Figure 1, for instance, the water molecule, with its positively charged hydrogen atoms and negatively charged oxygen atom, keeps reorienting again and again to follow the oscillating field.

That kind of repeated dipole rotation is resisted by inertia, intermolecular forces, friction and elastic restoring effects, so the dipoles cannot simply snap into alignment the instant the field changes. The “stubbornness” met during the constant reorientation turns electromagnetic energy into molecular motion, and the molecules gain kinetic energy. Then, as that kinetic energy accumulates across the bulk of the material, heat forms fairly uniformly throughout the volume, leading to fast heating that feels internal, pretty effective overall.

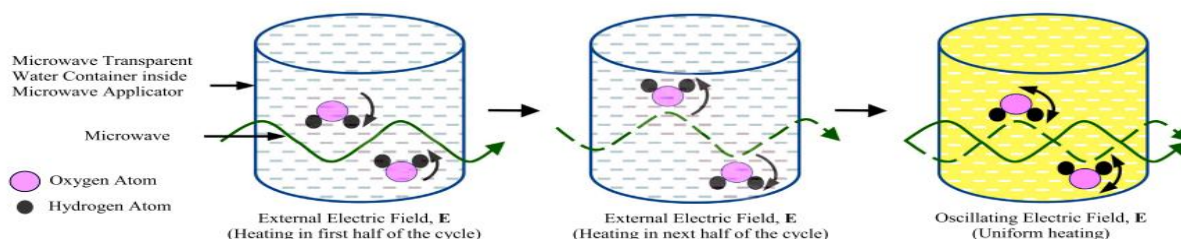


Figure 1. Heating mechanism in dipolar loss [9].

Conduction loss

Figure 2 shows, in a conduction based way, how microwaves heat things during processing. In particular, pure metals, metal matrix composites, and semiconductors like copper (Cu), aluminium (Al), silicon (Si), iron (Fe), and nickel (Ni) can lose a lot of microwave energy, mostly because they keep high electrical conductivity. Inside these materials there is a big crowd of free electrons (Fig. 2a), and once an alternating electric field, E is applied, they start to drift with an average velocity v (Fig. 2b). Since the electric field drops fast as it goes deeper into the conductive portion, an induced current (I_n) forms in a strong way (Fig. 2c). That current then gives rise to an induced magnetic field (H_n), which works against the magnetic field that was put in from outside. After that, the electromagnetic coupling kicks back on the travelling electrons, making them move back the other way with a reverse velocity (v_r). In other words kinetic energy gets pushed into the electrons while their movement is checked by inertial, elastic, frictional, and intermolecular forces, kind of together. And because the alternating electric field keeps flipping its direction over and over the whole process keeps repeating, continuously, so the material ends up producing heat efficiently, in a fairly uniform manner, and throughout its full volume, as can be seen in Fig. 2d [10,11].

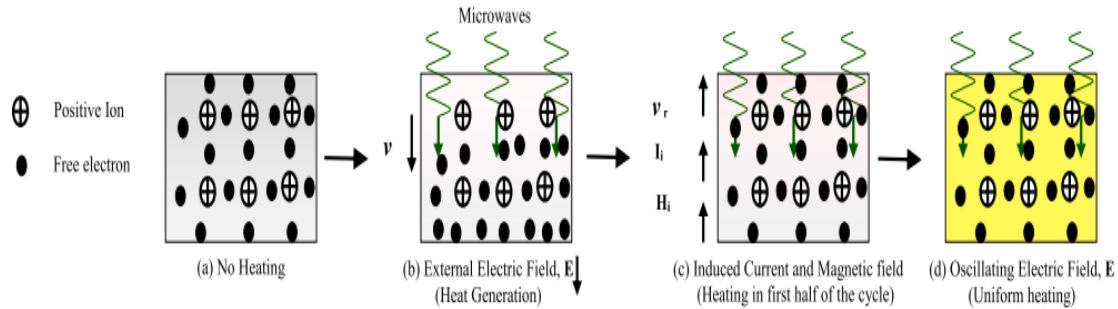


Figure 2. Heating mechanism in conduction loss [9].

2. Objective of study

To investigate and compare densification behavior, microstructure, surface integrity, and mechanical properties of Microwave Sintered and Conventional Sintered Magnesium, Vermiculite, and Tungsten composites in order to determine the efficiency and industrial feasibility of microwave sintering.

3. Research Methodology

The present investigation was carried out in order to investigate and compare the densification behavior, microstructure, surface integrity, and mechanical behavior of microwave sintered and conventionally sintered powder metallurgy composite of magnesium, vermiculite, and tungsten. The comparative experimental method was adopted wherein the same powder composition was subjected to both microwave sintering and conventional furnace sintering under similar processing conditions.

3.1 Materials

The initial materials used in this study were magnesium powder, vermiculite powder, tungsten powder, aluminium powder, copper powder, boron carbide (B4C), bentonite, and distilled water. Magnesium, vermiculite, and tungsten were used as the main matrix material to prepare different composite systems, whereas aluminium and copper were used to improve bonding and densification during sintering. Boron carbide was added as a reinforcement material to the magnesium composite due to its hardness and wear resistance. Bentonite was used as a temporary binder in green compact fabrication, and distilled water was used to ensure plasticity during powder mixing.

Six powder compositions were used during the experimental work. These included vermiculite, tungsten, magnesium, aluminium, copper, and boron carbide composites. Some of these powder compositions included 21gm vermiculite with additions of copper and aluminium, 30gm tungsten with additions of copper and aluminium, and 30gm magnesium reinforced by boron carbide with additions of aluminium and copper powder.

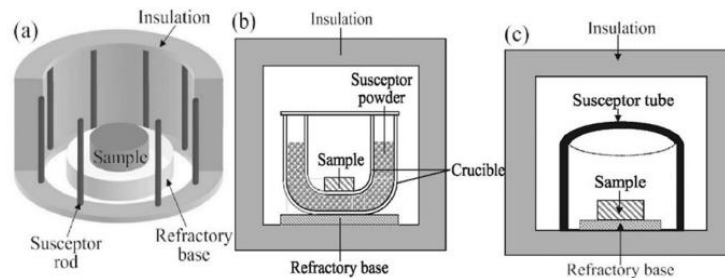


Figure 3. Schematic diagram of experimental setup [12,14].

3.2 Preparation of Green Compacts

Initially, all the raw powders were collected and dried to eliminate any absorbed moisture prior to mixing. The weights of the powders were measured as per the required compositions using the digital balance. The mechanical mixing continued till the uniform powder mix was attained. The bentonite and distilled water were introduced to enhance the green strength.

The uniform powder mixture was then compacted in a customized die-punch set to obtain the cylindrical green compact samples. Proper amount of compaction was applied to reduce the porosity without compromising the dimension of the samples. Then, after compaction, the green samples were dried for further sintering. The entire process of sample preparation involved collection of raw materials, mixing of powders, compaction, drying, and finally sintering.

3.3 Experimental Design

A three factor experimental design was used to study the effect of processing factors on the densification behavior. The processing factors chosen were the sintering temperature, the reinforcing powder percentage, and the sintering time. Levels of sintering temperature included 500°C, 700°C, and 900°C; levels of reinforcing powder percentage included 1.0%, 1.5%, and 2.0%; while the levels of sintering time included 3, 4, 5, and 6 min.

3.4 Microwave Hybrid Sintering

Sintering of the compact was performed using a laboratory microwave furnace at microwave hybrid heating. Since metal powders have a poor direct ability of absorbing microwaves, a microwave susceptor was used to induce heating as shown in Figure 3. In the current research, silicon carbide and charcoal powder were used as susceptor materials while alumina wool, refractory bricks, crucible made of quartz and alumina, and an atmosphere-controlled chamber were

introduced for better insulation and homogenous heat distribution.

The compacted specimens were placed into a ceramic crucible together with a microwave susceptor as illustrated in Figure 4. When heated by microwaves, the susceptor first absorbs the microwaves and produces heat that is further distributed throughout the compact. With rising temperature in the compact, volumetric heating results in rapid diffusion, particle bonding, and formation of the necks in the compact.

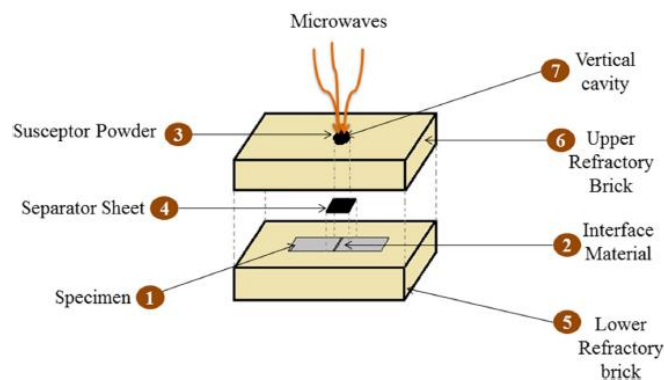


Figure 4. Schematic Diagram of Selective Microwave Hybrid Heating [13].

3.5 Conventional Sintering

To facilitate comparison, green compacts having the same composition were sintered using a conventional muffle furnace at comparable temperatures. In conventional sintering, the transfer of heat took place from the furnace walls to the specimen through conduction, convection, and radiation. This is unlike the microwave heating process, which used an internal heating method and resulted in comparatively greater temperature gradients and longer

sintering time. The conventionally sintered specimens provided references for the effectiveness of microwave-assisted sintering process.

3.6 Characterization Techniques

Several methods of physical and microstructural characterization were applied after the sintering process.

Bulk density and apparent porosity were calculated through the Archimedes principle. The dry weight, wet weight, and immersed weight were taken to calculate the densification and porosity values based on standard formulae. Density was the measure of good particle consolidation while porosity indicated void presence.

Microstructural analyses were carried out via optical microscopy at magnifications of 7X and 30X to compare the pore morphology, particle bonding, necking and phase distribution in microwave-sintered and conventional-sintered samples. Emphasis was placed on uniform distribution of pores, inter-particle bonding and homogeneity of the composite matrix.

Phase analysis and crystal structure analysis was carried out via X-ray Diffraction (XRD). The XRD patterns were used to identify the crystalline phases obtained after sintering process and the impact of variation in the reinforcement composition on the phase evolution during the sintering process. In addition, the intensity of the diffraction peaks and the phase composition of vermiculite-based and magnesium-based composites were also compared.

The integrity of the surface was assessed qualitatively via microscopic examination based on the occurrence of cracks, porosity connectivity, surface compactness and microstructure uniformity. In general, the experimental procedure made it possible to carry out a direct comparison of the microwave and conventional sintering processes since they involved the same materials composition and processing parameters except the heat source.

4. Results and Discussion

Performance of Microwave Hybrid Sintering (MHS) was assessed based on the densification, microstructural and phase development characteristics as compared with those produced by conventional furnace sintering (CFS). This assessment was conducted through measurement of density and porosity, microscopy studies, and XRD analysis. The findings show that microwave sintering provided better densification, less porosity and a more uniform microstructure than conventional sintering under comparable sintering conditions. This is due to the fact that the volume heating nature of microwave heating promotes diffusion and reduces thermal gradients during sintering.

4.1 Density and Porosity Analysis

Density and porosity are some of the key determinants of the effectiveness of sintering due to their direct representation of how much the particles consolidate in the process of heating. Density represents better diffusion and interparticle bonding, while low porosity represents effective removal of the cavities that have developed in the course of powder compaction.

The test results from various combinations of temperature, reinforcement amount, and sintering times show great disparity in density and porosity levels as illustrated in Table 1. Bulk densities ranged from approximately 5.21 to 7.88 g/cm³, while apparent porosities varied from 20.42% to 27.46% for the microwave sintered samples. The highest density was recorded at 900°C, 1.5–2.0% reinforcement, and 6 minutes holding time, while the relatively low density was recorded at lower temperatures and sintering times. This was also the case with the lowest porosities, which were attained at higher temperatures.

The increase in density as the sintering temperature increases is due to an increase in the diffusion of atoms between the neighboring powder particles. Increased temperatures lead to neck formation and grain boundary diffusion, which reduce the size of pores and hence increase particle consolidation. Similar results were obtained when increasing the sintering time from 3 to 6 minutes, as this allowed time for the pores to shrink due to diffusion.

It was found that there was always an inverse relation between density and porosity throughout all experiments carried out. Those samples having high densities had lower porosity levels. The graphical representation shown in the study confirms the inverse relationship between density and porosity. This means that increased densification was characterized by gradual elimination of the interconnected pores.

Microwave hybrid sintering improved densification compared with furnace heating, because in microwave heating the heat is generated from inside of the specimen instead of being transferred from the outside surface. Calculated value of density and average porosity value is illustrated in the graphical form Figure 5.

Table 1: Result of Density & Porosity with different combinations in case of Micro-Wave Sintering.

Sample No.	Inputs			Outputs	
	Temperature (°C)	Reinforcement Percentage (%)	Time (Min.)	Density (g/cm^3)	Avg. Porosity (%)
1	900	1.5	3	5.409	24.84
2	500	1.5	6	7.68	20.87
3	500	1	4.5	6.80	25.63
4	700	1	3	7.41	26.75
5	500	2	4.5	6.68	20.42
6	900	1	4.5	7.33	26.96
7	700	1	6	6.73	25.13
8	500	1.5	3	5.24	20.97
9	900	2	4.5	7.86	22.88
10	700	1.5	4.5	7.49	21.01
11	700	2	6	6.03	25.59
12	900	1.5	5	7.88	21.86
13	790	1.5	4.5	6.58	27.46
14	700	2	3	5.21	24.27
15	700	1.5	4.5	6.74	23.44

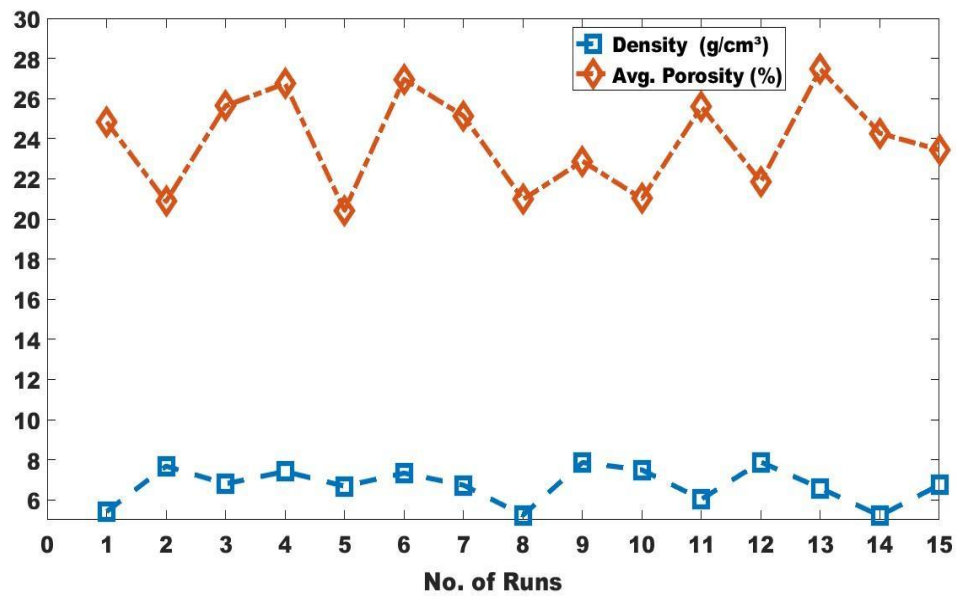


Figure 5. Experimental data of the density and porosity of the samples.

4.2 Microstructural Analysis

The optical microscopy was used to compare the microstructure of microwave and conventionally sintered samples at 7X and 30X magnifications. The comparison revealed that microwave hybrid sintering leads to a better quality of the microstructure in terms of homogeneity than the conventional one.

Table 2, shows the microwaved vermiculite-based composite (sample 4), there were quite fine and uniformly distributed pores in the matrix. There were no large pores clusters, which means there is a high degree of densification with elimination of all pores in the process of sintering. The boundaries of the particles were well connected, which means that there was an efficient neck growth with the bonding of the particles. The matrix looked continuous and homogeneous with less agglomerations of the reinforcing phase. On the contrary, the conventional sintered samples had bigger and non-uniformly distributed pores in their structure.

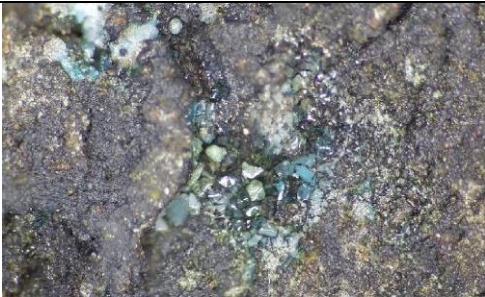
Table 2. Microwave Hybrid Sintering (MHS) vs Conventional Furnace Sintering (CFS) of Sample No. 4 (7X & 30X).

Sample Name	Magnification 7X	Magnification 30X
Sample 4 MHS		

Sample 4 CFS		
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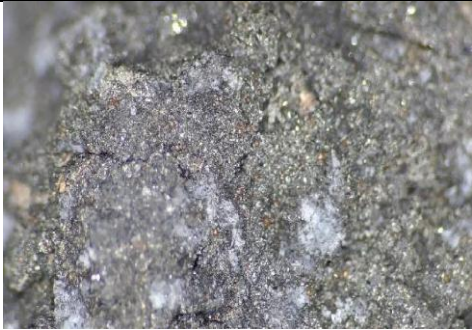
The same phenomenon was noted in the case of the tungsten-based composite (Sample 5) as shown in Table 3. In the case of microwave sintering, the samples had pockets of porosity which were isolated and well distributed throughout the matrix. The dense nature of the microstructure indicated strong bonding of particles and formation of necks between neighboring particles. The bright reinforcement phases were evenly distributed, signifying effective metallurgical bonding. On the other hand, the conventional sintering had interconnected pockets of porosity and irregular phase distribution due to inadequate bonding between neighboring particles.

Table 3. Microwave Hybrid Sintering (MHS) vs Conventional Furnace Sintering (CFS) of Sample No. 5 (7X & 30X).

Sample Name	Magnification 7X	Magnification 30X
Sample 5 MHS		
Sample 5 CFS		

The magnesium composite (Sample 6) further substantiated the superiority of microwave hybrid sintering process as shown in Table 4. The surface morphology of the sample that was sintered using microwave energy showed uniformity and smoothness along with distinct pores and improved particle intermixing. The change from one particle to another seemed to be smooth, showing faster diffusion and growth of necks between particles. The surface morphology of the other sample that was sintered by conventional method showed roughness, presence of interconnected pores and texture variations.

Table 4. Microwave Hybrid Sintering (MHS) vs Conventional Furnace Sintering (CFS) of Sample No. 6 (7X & 30X).

Sample Name	Magnification 7X	Magnification 30X
Sample 6 MHS		
Sample 6 CFS		

Microstructurally, the findings reveal the effect of microwave sintering on the process of accelerated diffusion, efficient pore removal, and enhanced homogeneity of the matrix. The process of rapid volumetric heating helps achieve simultaneous densification of the compact and minimizes microstructural heterogeneity.

4.3 X-ray Diffraction (XRD) Analysis

X-ray diffraction analysis was done to examine the crystal phase composition of microwave sintered composites as well as to assess the effect of the composition of the reinforcements on phase formation as illustrated in Figure 6. Diffraction patterns from Samples 4 and 6 after microwave sintering were taken.

In the case of the Al-Cu-Vermiculite composite, the number of diffraction peaks was smaller but with greater intensity compared to others. Diffraction peaks show that aluminum was the major crystalline phase formed after microwave treatment, along with other peaks belonging to copper phases and vermiculite phases.

However, on the other hand, the Al-Mg-B₄C composite showed more diffraction peaks, each with relatively low intensities. The increase in the number of diffraction peaks is attributed to the more complicated microstructure due to the reaction among magnesium, aluminium, boron carbide, and their reinforcement counterparts as a result of sintering. The higher variety of crystalline phases suggests increased interfacial reaction facilitated by the use of microwave heating with retention of the overall crystalline structure.

No signs of complete amorphization were observed in both the composites, indicating that microwave sintering has maintained the crystalline structure and facilitated phase formation. It can be deduced from the XRD results that reinforcement has an effect on the phase formation, diffraction peak intensity and the crystallography of the composite.

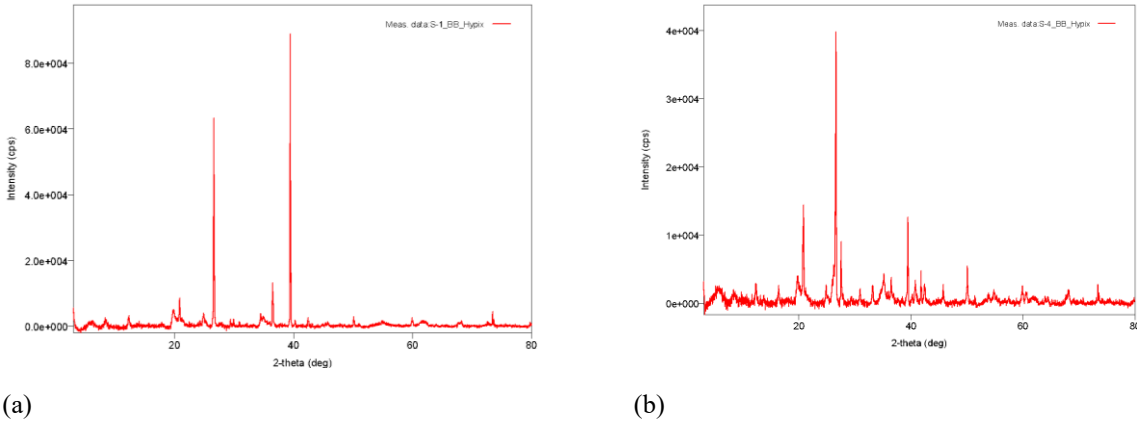


Figure 6. Shows the XRD Plot of (a) Sample 4, (b) Sample 6.

4.4 Comparison between Microwave and Conventional Sintering

It is evident that microwave hybrid sintering is more efficient than furnace sintering as far as densification and microstructure quality are concerned. Both processes were successful in terms of obtaining consolidated material samples; however, microwave treatment was more effective in ensuring proper densification, homogeneity of microstructure and surface quality, illustrated in Table 5.

The first advantage of the microwave treatment is related to its internal heating. Unlike conventional furnaces that provide progressive heat transfer from the external surface of the material towards its core, microwave technology enables volumetric heat generation throughout the volume of the sample. Consequently, this type of heating ensures faster diffusion process, neck formation between atoms, and prevents formation of any thermal gradients that might cause uneven shrinkage and porosity.

Microwave sintered samples had less porosity, better interparticle bonding and homogeneous phase distribution. This was confirmed by optical microscopy that showed less interconnected pores, less reinforcement agglomeration and smoother surface morphology.

On the other hand, the conventional sintering technique resulted in higher porosity, greater microstructural heterogeneity, particle interfaces, and formation of local clusters of pores. Due to low heating rate and heat transfer from the outside, diffusion was not efficient and led to the incomplete removal of pores and poor bonding of adjacent particles.

The practical benefits of the microwave hybrid sintering technique include reduced time of processing, lower energy costs, better uniformity of dimensions, higher material quality, and higher efficiency. These observations suggest that the use of the microwave sintering technique is preferable over the furnace one in the manufacture of magnesium-, vermiculite-, and tungsten-containing powder metallurgy composites for engineering purposes.

Table 5. Comparative assessment based on the available experimental observations.

Parameter	Microwave Heat Treatment	Conventional Heat Treatment	Support from experimental results
Density	Improved densification observed	Qualitatively lower	✓
Porosity	Finer pore distribution; residual porosity remains	Higher residual porosity reported	✓
Particle bonding	Better particle bonding in J4	Comparatively weaker bonding	✓

Microstructure	More compact and homogeneous in J4; heterogeneous in J5/J6	Rougher with agglomeration	✓
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5. Conclusion

The current research conducts a comparative analysis of microwave hybrid sintering and traditional furnace sintering techniques in relation to powder metallurgy composite materials of magnesium, vermiculite, and tungsten. The experimental analysis proves that the heating mechanism has a vital effect on the process of densification, pore reduction, microstructural formation, and phase development.

The results obtained from density and porosity measurements have confirmed the existence of the inverse connection between the discussed parameters, and the increase in sintering temperature and holding time leads to better densification. Microwave hybrid sintering was found to produce better density and less residual porosity due to the principle of volumetric heating and enhanced particle bonding. The maximal density of 7.88 g/cm³ and minimal porosity of 20.42% confirm the effectiveness of microwave sintering technique.

Moreover, micromorphology analysis revealed finer distribution of pores, strong interparticle bonds, better particle necking, and homogeneous structure in the case of microwave-sintered compacts whereas conventional sintering produces coarse pores, high microstructural heterogeneity and incomplete interparticle bonding because of the slow heating process. It can be concluded that microwave hybrid heating technique ensures better uniform energy distribution in the compact.

The success of crystallization of the composite phases was confirmed by X-ray diffraction studies following the microwave sintering process, which proved that the nature of the reinforcement material had a significant influence on the phase composition and diffraction peaks.

In summary, these results have confirmed that microwave hybrid sintering technology is a technologically viable and commercially feasible substitute to furnace sintering process for powder metallurgy fabrication technique. This new sintering technique has the potential of achieving higher levels of densification, improved microstructural uniformity, lower porosity, lower processing time and more efficient use of energy for the manufacture of advanced engineering products.

Future investigations should focus on quantifying the mechanical properties like hardness, compressive strength, wear characteristics, SEM and BET analysis of the microwave sintered composites.

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