

# Experimental Comparison of Microwave and Conventional Heat Treatment for Improving Surface Characteristics of Copper, Titanium, and Silicon Carbide Composites

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**Abstract:** Microwave heat treatment is emerging as an energy saving alternative to conventional thermal treatment owing to its fast volumetric heating, short processing time and enhancement in microstructural and mechanical properties of engineering materials. In this paper, the microwave heating efficiency of Copper (Cu), Titanium (Ti) and Silicon Carbide (SiC) composites by powder metallurgy is experimentally studied and compared with conventional heating methods. Composite samples were prepared by uniform powder mixing followed by compaction and thermal treatment using both microwave and conventional processing methods. To understand the effect of the heat treatment method on densification behaviour, microstructural changes and phase formation, the analysed samples were evaluated by density and apparent porosity metrics, optical microscopy and X-ray diffraction (XRD). The experimental results indicated that microwave heat treatment improved the adhesion of particles, pore distribution and densification compared to the specimens treated by conventional heat treatment. Optical microstructural analysis showed some microwave treated samples possessed fairly dense and uniform microstructures, whereas conventionally processed samples exhibited higher residual porosity and localised particle clustering. XRD analysis showed that the phase formation was strongly affected by the reinforcement composition and the Cu–SiC–Ti composites displayed more complex multiphase characteristics than the Cu–Ti composites after microwave processing. Although full densification was not achieved for the studied processing parameters, microwave heat treatment exhibited a great potential to improve the surface integrity and microstructural characteristics, while reducing the processing time. The results suggest that microwave heating can be a feasible alternative for processing advanced powder metallurgy composites, but further optimisation of processing parameters is needed for better densification and mechanical properties.

**Keywords:** Microwave and Conventional Heat Treatment; Copper alloy, Titanium and Silicon Carbide; X-ray diffraction analysis

## 1. Introduction

Powder metallurgy (PM) is an advanced manufacturing process widely used for the production of engineering components with accurate composition, near-net shape and tailored microstructural and mechanical properties [1]. The process involves the production or selection of metallic and ceramic powders, mixing of the powders to homogeneity, compaction of the mixture to green bodies and sintering to obtain metallurgical bonding and densification. Some advantages of the powder metallurgy process relative to the traditional casting and machining process are the better utilization of the material, better control of the composition, less production scrap, and the possibility of producing ceramic particle-reinforced metal matrix composites [2].



In the present study, Copper (Cu), Titanium (Ti) and Silicon Carbide (SiC) composite specimens were fabricated by powder metallurgy technique before heat treatment. The main reason for selecting powder metallurgy was the possibility of uniformly distributing reinforcement particles and to produce specimens with controlled initial porosity, which would allow a systematic comparison of microwave and conventional heat treatment. The properties of powder metallurgy products are greatly influenced by powder characteristics such as particle size, morphology, size distribution, apparent density, compressibility and chemical purity, which directly affect the green density, sintering behaviour and the final mechanical properties of the composites [3].

In the present study various composite formulations of Copper, Titanium and Silicon Carbide were prepared by mixing individual powders in some specific ratios. Bentonite was used as a bonding agent to enhance the green strength of the compacted samples. The mixing medium was distilled water to ensure homogenisation of the powder particles. Uniform blending is required to minimise particle segregation, attain high packing density and allow for uniform microstructural evolution during subsequent thermal processing [4].

The powder mixtures were blended and then pressed to produce green specimens having sufficient dimensional stability to be handled and thermally processed. The application of pressure during compaction increased the contact surface area between the neighbouring particles due to the mechanical interlocking and plastic deformation which in turn reduced the interparticle voids and improved the green density. The properties of the following densification during sintering are strongly dependent on the quality of the green compact. A higher green density usually leads to improved adhesion between particles and less residual porosity after heat treatment [5, 6].

In contrast to traditional research in powder metallurgy, mainly concentrating on the effect of compaction parameters, this research intends to evaluate the effectiveness of two different heat treatment techniques after specimen fabrication [7]. Green samples were compacted and then heated by microwave or conventional heating to investigate their effect on microstructural development, surface quality and mechanical properties. Microwave heating allows for volumetric absorption of the energy inside the material, resulting in rapid and relatively uniform internal heating. Conventional heat treatment relies on external heat transfer via conduction, convection and radiation. Differences in heating mechanisms are anticipated to affect diffusion behaviour, neck growth, densification and phase transformation during the sintering process [8].

Therefore, the powder metallurgy method used in this study provides a reliable experimental environment for the evaluation of the two heat treatment methods while ensuring uniform material composition and sample preparation parameters. Therefore, the observed differences in density, porosity, microstructure, particle adhesion, and phase evolution can be attributed to the heat treatment process rather than to differences in specimen preparation.

The study was aimed at comparison of the performance of microwave heat treated specimens with conventionally heat-treated specimens in terms of surface integrity, microstructural changes and mechanical properties to assess the effectiveness of microwave heat treatment in industrial processing of materials.

## **2. Objective of study**

To experimentally compare microwave heat-treated and conventionally heat-treated Copper-, Titanium-, and Silicon Carbide-based composites with respect to surface integrity, microstructural evolution, phase characteristics, density, porosity, and mechanical performance, thereby evaluating the suitability of microwave heat treatment as an alternative processing technique for advanced engineering materials.

## **3. Experimental Procedure**

### ***3.1 Materials***

The experiment was conducted with Copper (Cu), Titanium (Ti) and Silicon Carbide (SiC) powders as the main constituents of the composite. Bentonite powder was added as a binder to provide the strength to the compacted samples. Distilled water was used for the mixing process to make the constituent powder homogeneously dispersed.

Composites of six different compositions were made by changing the proportions of Copper, Titanium and Silicon Carbide in order to study the effect of microwave heating process and conventional heating process on their surface quality and mechanical properties. The prepared samples included SiC rich, Ti rich and Cu rich composites.

### 3.2 Sample Preparation

The unprocessed powders were then weighed based on their predetermined compositions and well-mixed with bentonite and distilled water to form homogeneous mixtures of the powders. The mixtures were then processed into green samples before heating. Among the representative compositions formed were Cu-SiC, Cu-Ti, and Cu-Ti-SiC composite materials with varying compositions. Six samples prepared from experiments for comparison will be discussed, considering the heating conditions of microwave heating and conventional heating.

### 3.3 Microwave Hybrid Sintering (MHS)

The heat treatment with microwave was done using the microwave furnace that had the refractory insulation system in the laboratory. Microwave hybrid heating system composed of refractory bricks, the alumina/quartz crucible, and charcoal powder as the susceptor were used in order to heat up the samples effectively. With the help of microwave hybrid heating system, the temperature was increased rapidly due to the volumetric heating of the compacted sample. The different microwave hybrid heating systems which include the rod type, powdered, and tubular susceptors for microwave heating system. Sintering has been done with the help of experimental design of temperature, reinforcement percentage, and holding time.

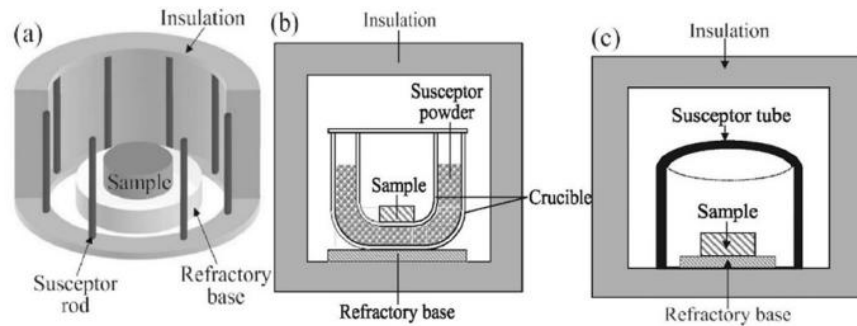


Figure 1: Schematic diagram for three different MHS with Rod-like (the picket-fence arrangement), Powdered (in the two-crucible set-up) and Tubular susceptor [8].

### 3.4 Conventional Furnace Sintering (CFS)

For the purposes of comparison, corresponding samples underwent thermal processing by means of conventional heating. Subsequently, both the microwave-heated and conventionally heated samples were examined via the same characterization processes. The comparison allowed the identification of the variations in densification, surface structure, particle bonding, porosity, and phase development in relation to the two heating methods.

### 3.5 Experimental Setup

The experimental set-up includes a microwave furnace, which was coupled with the refractory insulation, crucibles made of alumina/quartz, and charcoal powder as a microwave susceptor, along with sample holders. The process of powder mixture included sieving and mixing before specimen preparation. Design of Experiments (DoE) used three process parameters, namely temperature, reinforcement content, and sintering time. Three levels were chosen for each process parameter: the temperature range was 550-950°C, the reinforcement content was 1.0-2.0%, and sintering time was 3-6 min. Such process parameters were chosen to study their effect on the densification of the prepared composite specimens.

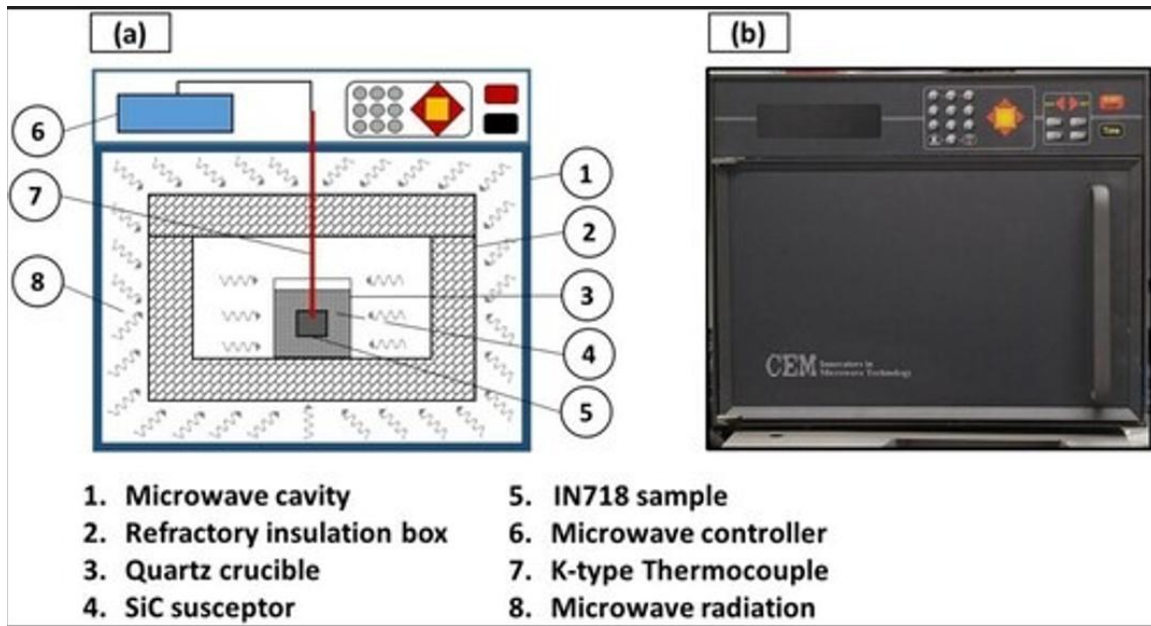


Figure 2: Experimental Set-up inside the Microwave furnace [8].

### 3.6 Characterization Methods

After heat treatments, the samples were analyzed to see the efficiency of microwave and conventional methods as illustrated in Figure 1 & 2. Density and apparent porosities were determined by means of experiments described in the research. The densification was characterized by means of mathematical calculations given in the equation below (Eq 1 and Eq 2). Optical microscopy analysis at magnifications 7X and 30X was used to determine the particle binding, pores distribution and microstructure uniformity. After the heat treatment, the phase formation was characterized by the X-ray diffraction analysis to identify the crystalline phases and compare the phase formation between various composite systems. Thus, the current paper focuses mainly on the comparative results of density, porosity, optical microscopy and XRD analysis.

The Sinterability or the tendency of the green compact particles to join together when heated is assessed through densification parameter (D.P.) which is expressed as:

$$\text{Bulk Density} = \frac{D}{(W - I)} \dots \dots \text{Eq}(1)$$

The porosity contents (  $\phi\%$  ) of the sintered specimens were found using equation:

$$\text{Apparent Porosity} = \frac{W - D}{W - I} \dots \dots \text{Eq}(2)$$

Where:

$D$  = Dry Weight,  $W$  = Wet Weight, and  $I$  = Soaked Weight

## 4. Results and Discussion

### 4.1 Density and Porosity Comparison

The densification properties of the composites based on Copper, Titanium and Silicon Carbide were studied by the measurements of density and porosity, after microwave heat treatment to improve the densification properties. Densification is a critical parameter for efficient sintering, as it reflects the reduction of internal voids, enhanced particle bonding, and the formation of a denser microstructure. The experimental results obtained under different

processing conditions showed considerable differences in density and porosity indicating that the microwave processing parameters have a great influence on the consolidation behavior of powder-based composites.

The experimental results showed that the density of the specimens varied significantly under the processing conditions studied as illustrated in Table 1. The recorded values were in the range of about 4.81 to 8.91 g/cm<sup>3</sup>. The average porosity was in the range of about 21.37% to 26.96%. These results show that the temperature, the ratio of the reinforcement and the sintering time have a high influence on the degree of particle consolidation during microwave sintering. The sample with the highest density also had a relatively reduced pore volume, which supports the inverse relationship between density and porosity.

For all experiments, density and porosity showed a consistent inverse relationship. With the progress of sintering, stronger metallurgical bonds were formed between adjacent powder particles, which reduced the interconnected pore channels and increased the bulk density. Experimental results show that full densification was not achieved for investigated conditions, because residual porosity persisted in all samples. This suggests that further optimisation of microwave processing parameters may be required to achieve near-theoretical density.

The densification behaviour is related to volumetric microwave heating, which causes a rapid absorption of internal energy inside the compressed powder particles. Microwave energy heats the entire sample uniformly, unlike conventional external heating, increasing atomic diffusion and improving particle adhesion. The augmented diffusion leads to pore shrinking, helps to form a neck between neighbouring particles, and gradually removes voids during the sintering process. Local thermal effects and different dielectric properties of the different constituent materials may also contribute to the residual porosity observed in some specimens.

Table 1: Result of Density & Porosity with different combinations in case of MHS.

|                   | <b>Inputs</b>           |                                     |                    | <b>Outputs</b>                         |                              |
|-------------------|-------------------------|-------------------------------------|--------------------|----------------------------------------|------------------------------|
| <b>Sample No.</b> | <b>Temperature (°C)</b> | <b>Reinforcement Percentage (%)</b> | <b>Time (Min.)</b> | <b>Bulk Density (g/cm<sup>3</sup>)</b> | <b>Apparent Porosity (%)</b> |
| 1                 | 900                     | 1.5                                 | 3                  | 6.409                                  | 23.34                        |
| 2                 | 500                     | 1.5                                 | 6                  | 7.08                                   | 21.84                        |
| 3                 | 500                     | 1                                   | 4.5                | 5.10                                   | 24.13                        |
| 4                 | 700                     | 1                                   | 3                  | 8.91                                   | 25.95                        |
| 5                 | 500                     | 2                                   | 4.5                | 6.28                                   | 21.72                        |
| 6                 | 900                     | 1                                   | 4.5                | 6.99                                   | 26.96                        |
| 7                 | 700                     | 1                                   | 6                  | 6.73                                   | 24.93                        |
| 8                 | 500                     | 1.5                                 | 3                  | 4.81                                   | 21.37                        |
| 9                 | 900                     | 2                                   | 4.5                | 6.56                                   | 23.74                        |
| 10                | 700                     | 1.5                                 | 4.5                | 6.49                                   | 22.01                        |
| 11                | 700                     | 2                                   | 6                  | 6.03                                   | 24.71                        |
| 12                | 900                     | 1.5                                 | 6                  | 7.88                                   | 23.86                        |
| 13                | 700                     | 1.5                                 | 4.5                | 5.19                                   | 26.45                        |
| 14                | 700                     | 2                                   | 3                  | 5.94                                   | 23.27                        |
| 15                | 700                     | 1.5                                 | 4.5                | 7.14                                   | 24.75                        |

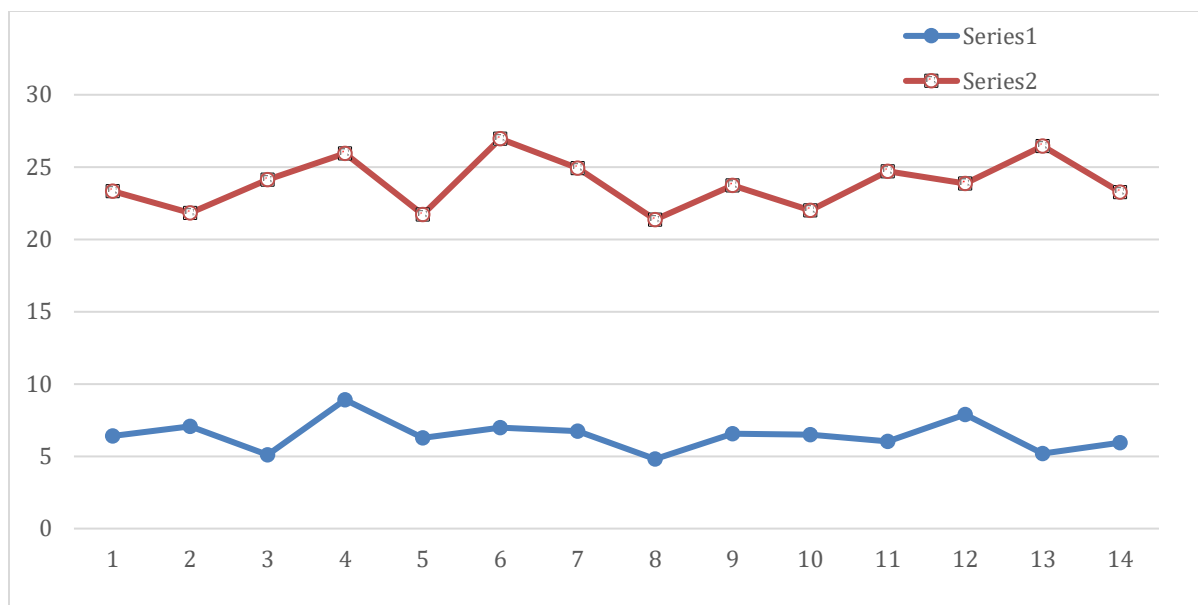


Figure 3: Experimental data of the density and porosity of the samples.

Figure 3 shows the graph, series 1 (blue line) represents density and series 2 (orange line) represents apparent Porosity.

#### 4.2 Optical Microstructure Comparison

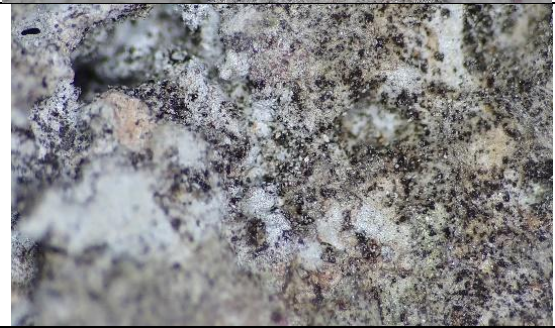
Optical microstructural analysis was carried out with an optical microscope at 7X and 30 X magnifications. The 7X images give an overview of the general surface morphology and pore distribution, whereas the 30X images allow for a more detailed analysis of particle bonding, neck formation and localised microstructural features. Comparison micrographs reveal significant differences in particle adhesion, pore arrangement and overall structural homogeneity for both processing methods.

In the sample 4 after microwave treatment, the microstructure was relatively dense and homogeneous. It appeared that the particles were more effectively interlinked, with smaller and more homogeneously distributed pores in the matrix. Such characteristics indicate better densification during microwave processing as the volumetric heating facilitates diffusion between adjacent particles. The reduction in pore size and the increase in particle interaction suggest that the microwave energy contributed to a more efficient neck formation during sintering, thus improving the structural integrity.

Table 2, the surface of the sample 4 treated by conventional heat treatment was much rougher, with higher residual porosity and places of particle clustering. More prominent pore clusters were observed, indicating relatively less densification and insufficient particle coalescence in normal heating conditions. The results support the claim that microwave thermal processing has led to better microstructural properties for this specimen.

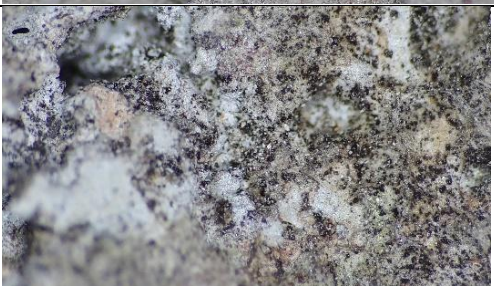
Table 2. Microwave Hybrid Sintering (MHS) vs Conventional Furnace Sintering (CFS) of Sample No. 4 (7X & 30X).

| Sample Name | Observation Magnification 7X | Observation Magnification 30X |
|-------------|------------------------------|-------------------------------|
|-------------|------------------------------|-------------------------------|

|                         |                                                                                   |                                                                                    |
|-------------------------|-----------------------------------------------------------------------------------|------------------------------------------------------------------------------------|
| <b>Sample 4<br/>MHS</b> |  |  |
| <b>Sample 4<br/>CFS</b> |  |  |

The results regarding sample 5 indicate that after microwave and conventional heating the samples maintained high surface roughness and residual porosity as represented in Table 3. The microwave treatment caused more noticeable local variations in surface texture due to rapid volumetric heating and localised absorption of microwave energy. These local thermal effects lead not only to nonuniform surface characteristics, but also indicate that the internal heating was still in progress during the whole sintering process. The specimen traditionally processed was seen to have a more uniform appearance. Both specimens showed significant clumping and lack of adequate particle adhesion.

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

| <b>Sample Name</b>      | <b>Observation Magnification 7X</b>                                                 | <b>Observation Magnification 30X</b>                                                 |
|-------------------------|-------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|
| <b>Sample 5<br/>MHS</b> |  |  |
| <b>Sample 5<br/>CFS</b> |  |  |

For sample 6 a similar trend was observed, with both samples showing uneven surfaces and clear residual porosity. Surface morphology was more heterogeneous in the microwave treated specimen than the more uniform

appearance in the conventionally processed specimen shown in Table 4. However, both samples showed residual pores and particle clustering indicative of incomplete densification under the processing parameters investigated.

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

| Sample Name  | Observation Magnification 7X                                                       | Observation Magnification 30X                                                       |
|--------------|------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|
| Sample 6 MHS |   |   |
| Sample 6 CFS |  |  |

In general, optical microstructural analysis shows that microwave processing improves particle bonding and reduces pore size in some composite systems. However, the microwave sintering results appear to be dependent on material composition and processing parameters since full densification has not been achieved for all the specimens studied.

#### 4.3 XRD Analysis

X-ray diffraction analysis was performed to study the crystalline phases formed after microwave heat treatment and to study the effect of reinforcement composition on phase evolution. The study shows the XRD patterns of typical composite samples sintered by microwave, as well as a qualitative analysis of the diffraction characteristics observed as observed in Figure 4, (a) shows for the sample 4 and (b) shows for the sample 6.

For Cu–Ti composite, it showed small amount of strong and sharp diffraction peaks which indicated that the dominant phase after microwave treatment was crystalline copper. The relatively lower phase complexity is suggested by a limited number of diffraction peaks, indicating that the primary components retained their crystallographic identity throughout the thermal processing.

On the other hand, the Cu–SiC–Ti composite showed a larger number of diffraction peaks with relatively less individual intensities. The increase in peak density shows the development of a more complex multiphase microstructure. As per the results, the higher number of diffraction peaks indicates stronger interactions between Copper, Titanium and Silicon Carbide in microwave sintering, which leads to better phase formation in the composite material.

The differences in diffraction properties indicate that the composition of the reinforcement is an important factor in the development of phases during microwave processing. The composite with SiC showed more

crystallographic complexity than Cu–Ti composite, which is a reflection of the enhanced interfacial interactions between the reinforcement particles and the matrix. These results support the contention that microwave heating can trigger phase evolution and promote microstructural modification in multiphase composite materials.

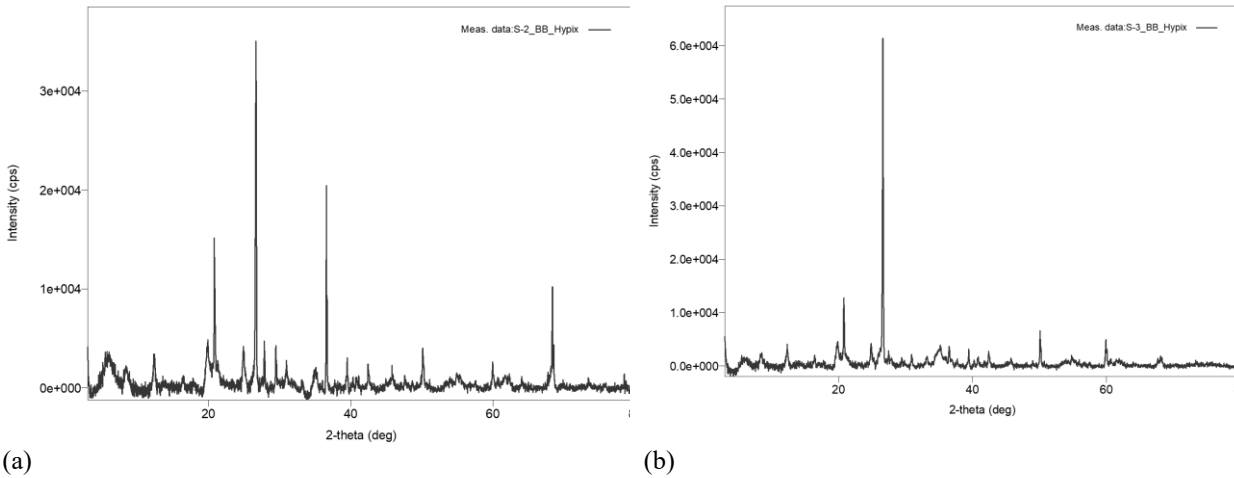


Figure 4: Shows the XRD Plot of (a) Sample 4, (b) Sample 6.

#### 4.4 Overall Comparison between Microwave and Conventional Heat Treatment

On the basis of the observations made in the experiment, the microwave heat treatment was observed to be relatively effective in improving the bonding of particles, providing fine pore distribution and better densification than the conventional heat treatment illustrated in Table 5. Nevertheless, residual porosity was still observed in the materials fabricated using either of these two processes, which means that the process did not lead to full consolidation of the materials under the selected experimental conditions. Some of the materials treated with the microwave heat treatment were characterized by denser microstructure while the conventional heat treatment led to relatively rough surfaces. The XRD analysis confirmed the formation of phases, especially in the Cu–SiC–Ti composite system.

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 K4                              | Comparatively weaker bonding      | ✓                                 |
| Microstructure   | More compact and homogeneous in K4; heterogeneous in K5/K6 | Rougher with agglomeration        | ✓                                 |

## 5. Conclusion

This research was aimed at assessing the effectiveness of microwave heating technique in comparison with conventional heating techniques for copper, titanium, and silicon carbide-based composites made through the powder metallurgy process. The study showed that microwave heat treatment increased the densification, bonding of particles, and also results in a better pore distribution than the conventional process. The optical microstructure revealed relatively dense structures, while the XRD results showed the presence of crystalline phases, revealing the effect of reinforcements on the development of different phases. However, despite the presence of pores in the samples, the

results have shown that microwave heat treatment is an effective processing technique for enhancing surface properties of powder metallurgy composite materials.

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