



## Sintering and Characterization of SiC Reinforced Ni Powders in Microwave Furnace

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### Abstract

In this study, SiC reinforced nickel matrix composites, microwave at different temperatures It is produced by sintering in oven. Nickel deposition on SiC powders was achieved by using electroless nickel plating technique. It is sintered at temperatures between 500°C, 600 °C, 700°C, 800°C and 900°C under Ar atmosphere. XRD, SEM (Scanning Electron Microscope), pressure test and hardness measurements were used to characterize the properties of sintered samples in microwave furnace. The experimental results, maximum compressive strength ( $\sigma_{max}$ ) and hardness (HV) were carried out at 800 °C. Microwave sintering of Ni-coated SiC powders without electric current can be a promising technique for producing ceramic-reinforced nickel composites.

### Key Words

*“Microwave sintering, Powder metallurgy, Composites, Nickel plating”*

## 1. Introduction

Composite materials are being used with increasing speed in many advanced engineering applications (Yönetken 2018; Tang et al. 2003; Zang et al. 2005; Wu et al. 2014; Jarzabek et al. 2017). Powder Metallurgy is a more advantageous production technique compared to other production methods in terms of producing high-tech materials, high strength starting from powdered raw materials, less error, cheaper processes and providing physical and chemical properties of desired powders (Malecki and Micek-Ilnicka 2000; Söyler 2007). Electroless plated nickel plating has been widely used in many areas. Because of electrolysis plating technique, non-homogenous coating is achieved. High hardness, abrasion resistance and corrosion resistance high performance products were obtained in Ni coated composite samples. Electroplating Ni coating is a simple process with low cost. It can also be used in the surface coating of particles with a complex shape. This feature has been preferred in the industry coating method. (Zhang et al., 2015).

Ceramic-Metal composites reinforced with silicon carbide (SiC) are a well-known and commonly used material in applications. However, SiC is susceptible to SiO<sub>2</sub> conversion very quickly in the oxygen environment due to its poor condensation and high decomposition. Due to the high cost of produced samples, its commercial success is limited (Mandal et al., 2001). Microwave furnaces (MWs) are electromagnetic waves operating at frequencies ranging from 300 MHz to 300 GHz, and wave frequencies ranging from a few cm to several mm. Unlike conventional heating, the penetration of microwave signals into materials causes a volumetric distributed heat source. As a result, the microwave sintering method enables faster heating of both small and large samples compared to conventional heating (Chatterjee et al., 1998).

Silicon carbide, which has many good properties such as good thermal conductivity, electrical conductivity, chemical stability, high mechanical strength and low friction, can be used as both structured materials and functional materials (Chen, et al., 2003).

SiC-containing ceramic metal composites have higher thermal properties and chemical stability (Somiya et al., 2003).

SiC powder interacts with microwave frequency radiation at ambient temperature. The energy of the electromagnetic field changes into heat energy (Horatiu et al., 2007).

In this study, the production of SiC carbide doped ceramic-metal composites and production of the composite was performed using microwave sintering technique. Composites produced by applying physical and mechanical tests to the produced samples were characterized.

## 2. Preparation of Sample

### 2.1 Material

In this study, two different aspects were used. Firstly, SiC powders and Ni powders were mixed in a constant speed mixer for 24 hours to ensure a homogeneous mixture. Silicon carbide (SiC), ceramic powder and metal as Nickel (Ni) powder. SiC powders of 10 µm and 99.5% purity and Ni powders of 3 µm and 99.5% purity were used, both of which were obtained from Johnson Matthey Materials Technology. The aim of this study is to reinforce SiC ceramic powders with Ni. Pure nickel was obtained from which Ni powders can be added directly in the mixture or can be obtained by coating with nickel chloride (NiCl<sub>2</sub>.6H<sub>2</sub>O) which is used in the electroplating nickel plating bath (Sverdel et al., 1995) (Changhong, et al., 1997).

### 2.2 Method

In the experimental study, samples were prepared by two different methods. In the first method, the homogeneous mixture obtained by mixing SiC-Ni powders for a 24 hour period was formed as cold under 300 bar in the uniaxial hydraulic press and made ready for sintering. In the second method, silicon carbide powders were deposited on Ni SiC powders using electroless nickel plating technique. Then, it was shaped under uniaxial hydraulic press under pressure of 300 bar. The shaped raw samples were sintered in the industrial microwave sintering furnace for one hour in the temperature range of 500-900°C under an atmosphere of argon gas. Sintered samples were sanded and polished for mechanical and metallographic analysis. 20% SiC powders were used by weight, Nickel Chloride, Ammonia, Hydrazine hydrate and pure water were used in the Ni-bath without electricity. The content of the coating bath is given in Table 1.

**Table 1.** The chemicals of Nickel plating bath and their ratios

Chemicals	Ratio
Silicon Carbide (SiC)	6
Nickel Chloride ( NiCl <sub>2</sub> .6H <sub>2</sub> O)	96
Hydrazine Hydrate (N <sub>2</sub> H <sub>4</sub> .H <sub>2</sub> O)	20%
Distilled Water	80%
Temperature (°C)	90-95°C
pH Value	9-10

Nickel plated silicon carbide powders were purified from chemicals by washing with pure water after plating and made ready for subsequent processing. The experiment flowchart is shown in Figure 1

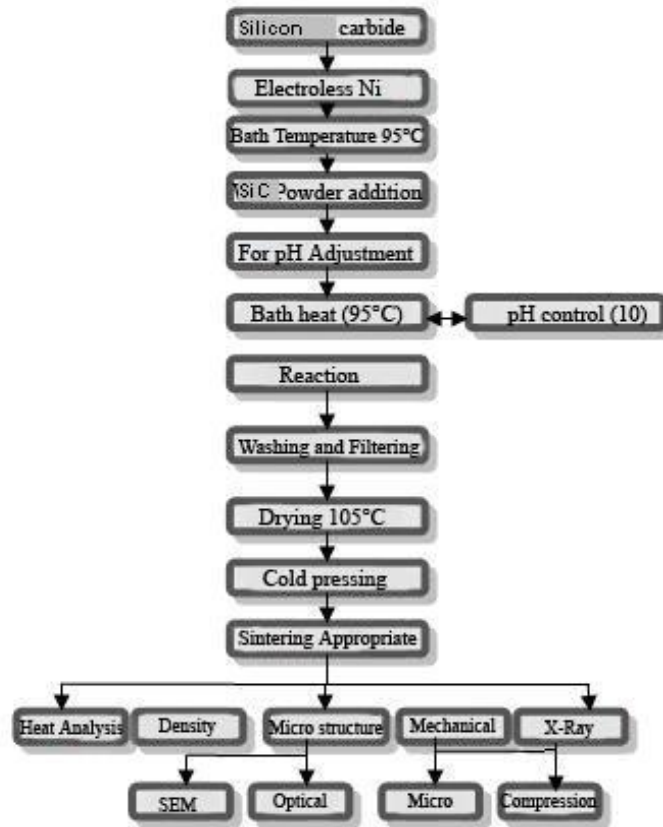


Figure 1. Experimental study flow diagram

In order to use sintering, Industrial sintering furnace was used. Furnace Phoenix brand 2450MHz 1500W 230V has label values. Sintering furnaces were produced in microwave sintering furnace at temperatures of 500-900 ° C for 1 hour. SEM-EDX analyzes were performed after sanding and polishing the sintered samples. SEM photographs were taken with LEO143OVP Röntech device. In addition, a Shimadzu-AG / IS 100kN tester was used to measure the compressive strength of the samples.

Micro hardness of the samples was measured with the Shimadzu HMV 2 L micro hardness tester. The hardness values were determined by taking the average of the hardness values taken from 10 different areas for each sample in the micro hardness measurements applied to the samples.

### 3. Experimental Findings

In this study, the samples prepared and shaped by two different methods were sintered in the industrial microwave sintering furnace at temperatures ranging from 500°C to 900°C. The samples were tested by making them ready for physical, mechanical and metallographic analyzes. In the study, the samples prepared and shaped (pressed) through two different methods were sintered at temperatures ranging from 500°C to 900°C in microwave furnace and made ready for physical, mechanical and metallographic analyses.

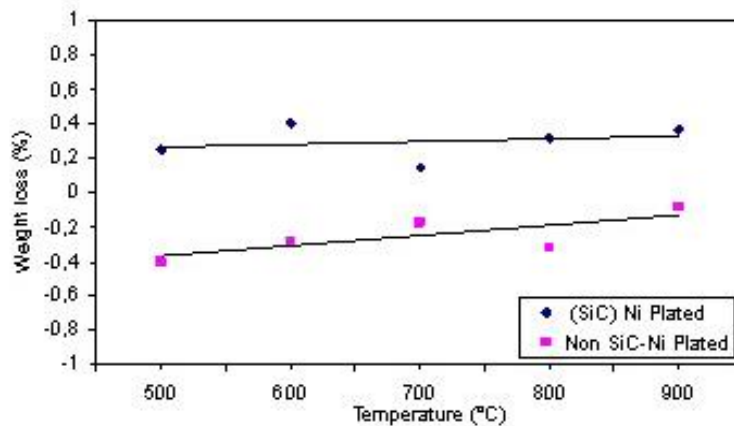


Figure 2. Percent weight change in the sintered samples depending on the temperature

Density-temperature change graphic is shown in Figure 3. The pre-sintering raw density of the plated sample was estimated to be 4.21gr/cm<sup>3</sup>. The highest post-sintering density was achieved at 700°C as 4.57gr/cm<sup>3</sup>. The highest post-sintering density of the non-plated sample was obtained at 700°C. The pre-sintering density of the non-plated density was estimated to be 4.85gr/cm<sup>3</sup>. The highest post-sintering density was observed to be 5.34gr/cm<sup>3</sup> at 700°C.

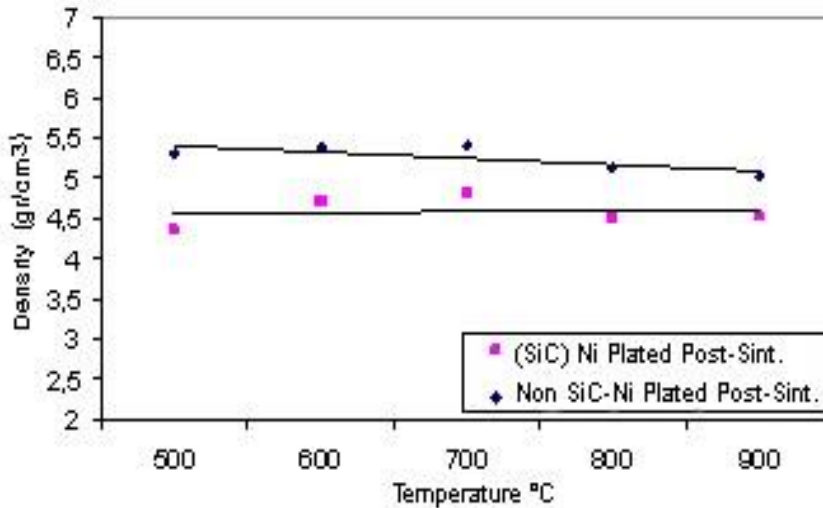


Figure 3. The graphic of density change depending on the temperature

**3.1 Analysis of the Physical Properties of the Samples**

In this study, Two different methods were used to produce SiC reinforced composite samples in composite production. pre and post sintering density of the samples were determined. Figure 2 is a graph showing the weight change of samples produced by a mixture of sintered samples and powders by coating depending on the temperature. Ni weight loss occurred in the samples produced by coating and weight increase occurred in the uncoated sample. This showed that uncoated samples were more easily oxidized and that sintering was not good despite the use of argon atmosphere.

**3.2 Analysis of the Mechanical Properties of the Samples**

The compression strength and microhardness of the ceramic-metal composite material produced by powder mixing and non-electric Ni coating methods were measured. The relationship between the sintering temperatures and compression strength values of the produced samples is given in Figure 5.

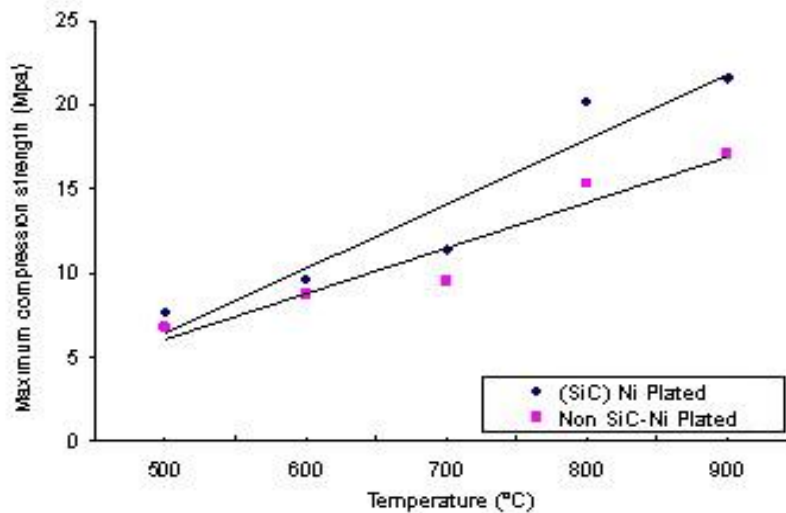


Figure 5. Compression strength test applied to the samples sintered at different temperatures

The microhardness-temperature change graph of the samples produced by mixing the powders and using the electro-current Ni coating methods is given in Figure 6. Micro hardness values of the produced composite samples, SiC powders were coated with Ni coating method without electric current. Produced samples were made using microwave sintering furnace at temperatures between 500-900 ° C. Samples produced using SiC-Ni powders and the Electroless Ni coating method were subjected to mechanical tests after sintering at 500-900 ° C.

The highest micro hardness value of the composite samples produced by using the Ni-Finite method without electric current was measured as 136HV at 900 ° C. Microhardness of samples produced by using electroless Ni plating method gave higher results than mixing of SiC-Ni powders. The highest micro hardness of the samples produced by using electroless Ni plating method was measured as 148HV in sintered composite samples at 800 ° C.

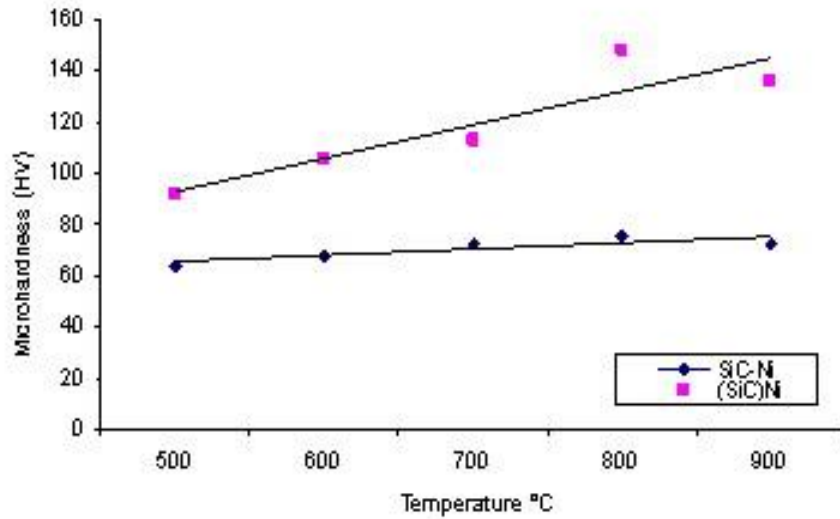


Figure 6. Microhardness - Temperatures curve of different product method.

### 3.3 Metallographic Analysis

Specimens after the sintering of SiC-Ni powders and the samples produced by using electroless Ni plating method, surface polishing processes were performed. After the electroless nickel plating process, it was examined by applying SEM EDX analysis whether Ni coating was applied to the powders. Fig. 7 shows the enlarged SEM images of ceramic-metal composite samples obtained from non-electric Ni-coated (SiC) powders and sintered at 800 ° C at different scales. In Figures 7 a and b, it was observed that the particles were better bonded to each other in the Ni (SiC) composite sample and the particles were larger. In the SEM image, there were pores with homogenous distribution between the grains.

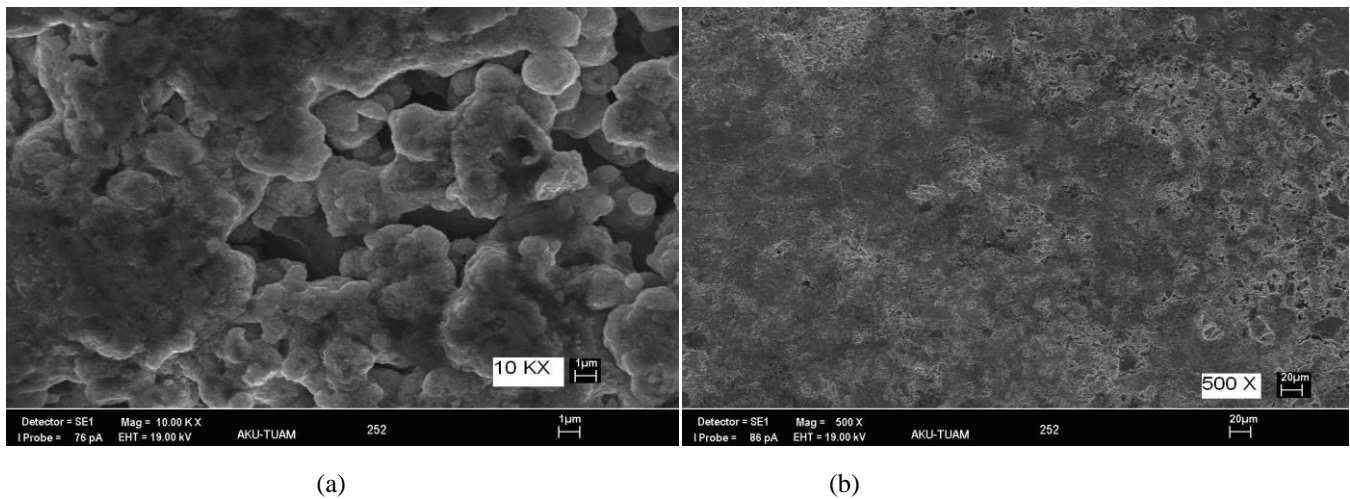
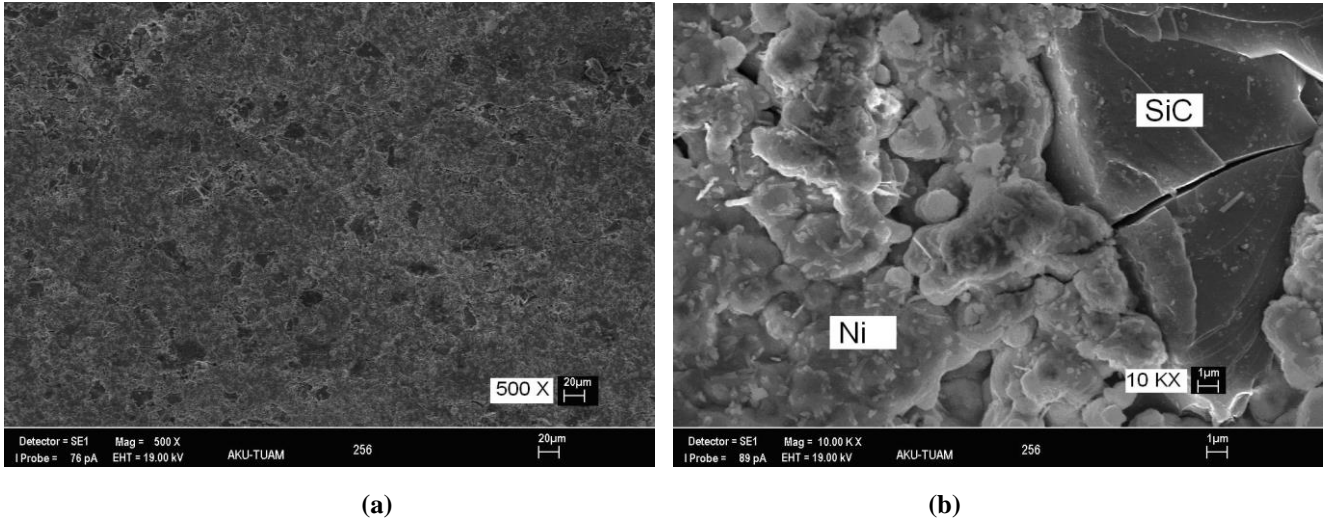


Figure 7. SEM view of (SiC)Ni composite at 800°C

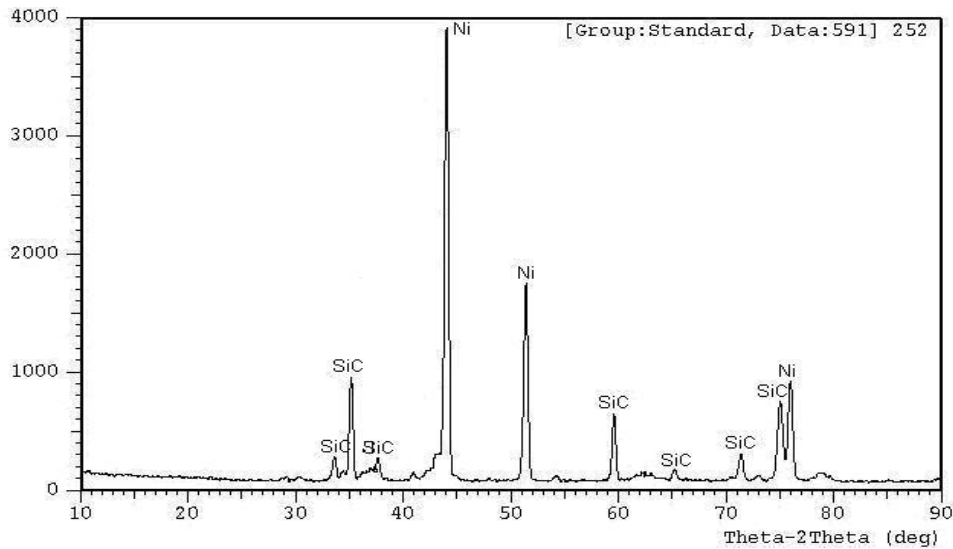
The SEM images of the composite samples produced by sintering at 900 ° C by mixing the SiC and Ni powders are given in Figure 8a-b. Figure 8a shows the SEM image of the WC-Ni composite at 500X magnification. In Figure 8b, the 10kX magnified SiC particle is clearly visible..



**Figure 8.** SEM view of SiC-Ni composite at 900°C

### 3.4 XRD Analysis

In Figure 9, shows the peaks of SiC and Ni phases. In the sintered composite sample (SiC) at the microwave oven at 800 ° C the XRD graph of the Ni composite is given. Since the sintering takes place at low temperature, no different phase is formed.



**Figure 9.** The XRD graphic of Nickel Plated SiC composite Sintered in Microwave Furnace at 800°C

## 4. Results & Discussion

As a result of the mechanical and metallographic tests applied to SiC-Ni composite samples, the following findings were obtained.

- In samples sintered at 900 ° C, the average hardness value of the Ni-coated SiC after sintering was measured as 89.85HV.
- The highest compressive strength was obtained as 22.24MPa at 900 ° C (Fig. 5).
- The highest density of composite samples produced in sintered Ni coated SiC powders at different temperatures was 4.57gr / cm<sup>3</sup> at 700 ° C (See Figure 1). In the coated samples, the pre-sintering density was calculated to be 4.21 g / cm<sup>3</sup>.
- The density before sintering in uncoated samples was calculated as 4.85gr / cm<sup>3</sup>. The highest density after sintering was 5.34 g/cm<sup>3</sup> at 700 ° C.
- In the composite samples produced by using electroless Ni plating method, the highest micro hardness was measured as 148HV at 800 ° C. The highest microhardness values of the samples produced by mixing the powders were measured as 37.8HV at 700 ° C.
- It has been determined that composite samples produced by using electroless Ni coating method have more homogeneous microstructures and less pores.

It has also been found that the mechanical properties of the coated samples produced using the electroplating Ni coating method are higher than those of the uncoated samples.

It has been concluded that SiC powders give a positive result to Ni-coating and microwave sintering method is more advantageous than classical sintering technique because of temperature, duration and low energy consumption.

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