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PAGES: 223-228

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MICROWAVE SINTERING OF ELECTROLESS Ni PLATED B_4C POWDERS

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ABSTRACT

Nickel matrixes reinforced with B_4C have been fabricated by microwave sintering at various temperatures. A uniform nickel layer on B_4C powders was deposited prior to sintering using electroless plating technique, allowing close surface contact than can be achieved using conventional methods such as mechanical alloying. The reactivity between B_4C powders to form compounds is controlled through Ni layer existing on the starting powders. A composite consisting of quaternary additions, a ceramic phase, B_4C, within a matrix of Ni B_4C and etc., has been prepared at the temperature range 500°C-900°C under Ar shroud. XRD, SEM(Scanning Electron Microscope), compressive testing and hardness measurements were employed to characterize the proerties of the specimens. Experimental results carried out for 600°C suggest that the best properties as σmax and hardness (HV) were obtained at 600°C and the microwave sintering of electroless Ni plated B_4C powders can be used to produce ceramic reinforced Nickel composites.

Keywords: Microwave Sintering, Powder Metallurgy, Ceramic-Metal Composites, Electroless Nickel Plating

1. INTRODUCTION

The microwave sintering ceramics and hardmetals technology is a prospective industrial technology which will have great advantages in a short time sintering and uniform heating [1]. Microwave heating is method having do self-heating from the internal by absorption of microwave power. Therefore, the rapid heating is possible compared with external heating by thermal conduction or radiation. When this method is utilized in processing of ceramics, the fine grain, uniformity and high densification are expected, and electric and mechanical properties can be improved [2]. Electroless plating is a chemical reduction process, which depends on the catalytic reduction of a metallic ion from an aqueous solution containing a reducing agent, and the subsequent deposition of the metal without the use of electrical energy. [3]. The use of microwave energy for materials processing has major potential, and real advantages over conventional heating. These include: Time and energy savings, Rapid heating rates, Considerably reduced processing time and temperature, Fine microstructures and hence improved mechanical properties and better product performance, Lower environmental impact [4].

Metallic materials with small grains exhibiting a high strength are interesting from both theoretical and experimental point of view. Further enhancement of their mechanical properties is possible due to reinforcement by ceramic particles [5].

Electroless plating is at the present time a commonly used industrial technique for deposition of metal coatings. Electroless deposition is a very simple process which can be used to obtain amorphous metallic coatings of uniform thickness on metallic or non-metallic substrates. Useful electroless deposits have been produced of nickel, cobalt, palladium, copper, gold, silver and alloys involving one or more of these metals. Particularly, electroless nickel coatings are widely used to protect surfaces of engineering metal components, against degradation by corrosion and wear or to build up of worn areas [6]. For industrial processes such as heating by microwave is preferred in order to reduce processing time and lower cost of energy [7]. Microwave radiation is considered to be in the range 300MHz and 300GHz, which is well above UV range with higher wavelength and lower energy levels [8].

In this study, the ceramic-metal composites were obtained by using electroless nickel (Ni) plating with boron carbide (B_4C) powders.

Ceramic materials, which include monolithic ceramics and ceramic-matrix composites, have been identified as potential candidates for high-temperature structural applications because of their high-temperature strength, light weight, and excellent corrosion and wear resistance.

2. MATERIAL-METHOD AND PREPARATION OF SAMPLE

2.1. Material

In this study, Boron carbide (B_4C) was used as ceramic powder and Nickel (Ni) powder as metal. B_4C powders with 10µm grain size and 99.5% purity which was both provided from Johnson Matthey Materials Technology Company, was used. The aim of this study was to reinforce B_4C ceramic powders with Ni. It was thought that Ni powders can either be added in the mixture directly or obtained through plating with nickel chloride ($NiCI_2.6H_2O$) used in electroless nickel plating bath [6].

2.2. Method and Preparation of Sample

In the experimental study the samples were prepared through two different methods. In the first method, homogeneous mixture obtained through mixing B_4C-Ni powders for a day was shaped in hydraulic press coolly under 200 bar and made ready for sintering. In the second method, Boron carbide powders were plated using electroless nickel plating technique and then shaped in hydraulic press again coolly under 200 bar pressure. The shaped samples were sintered for an hour within the temperature range 500-900ºC under argon gas atmosphere in microwave furnace. The sintered samples were made ready for mechanical and metallographic analyses. In electroless Ni plating bath, 20% B_4C powders, Nickel chloride, Ammoniac, Hydrazine hydrate and distil water by weight were used. The contents of the plating bath are given in Table 1.

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

Chemicals	Weight(gr)
Boron Carbide (B_4C)	21
Nickel Chloride ($NiCI_2.6H_2O$)	36
Hydrazine Hydrate ($N_2H_4.H_2O$)	20
Distil Water	94
Temperature (°C)	95°C
pH Value	10

Nickel plated silicon carbide powders were purified from chemicals by washing with pure water after plating and made ready for subsequent processing

Sintering was performed at 500-900°C in Phoenix brand 2450MHz 1500W 230V microwave furnace. SEM-EDX analyses were employed on the sintered samples. SEM photographs were taken with LEO143OVP Röntech device. Furthermore, Shimadzu-AG/IS 100kN testing device was used to measure the compression strength of the samples and the microhardnesses of the samples were measured with Shimadzu HMV 2 L microhardness device. The microhardness measures were obtained by taking the mean of hardness values taken from 10 different areas for each sample.

3. EXPERIMENTAL FINDINGS

In the study, the samples prepared and shaped (pressed) through two different methods were sintered at temperatures ranging from 500ºC to 900ºC in microwave furnace and made ready for physical, mechanical and metallographic analyses.

3.1. Analysis of the Physical Properties of the Samples

The pre-sintering and post-sintering densities of the samples prepared using two different methods were determined.
In Figure 1, the percent weight change graphic for plated and non-plated samples depending on the temperature is shown. In the plated sample percent weight loss took place whereas increase by weight occurred in the non-plated sample. This situation shows that oxidation took place in the sample and sintering was not at the desired level.

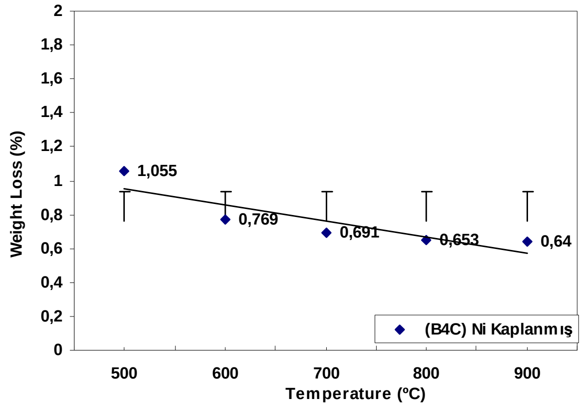


Figure 1. Percent weight change in the sintered samples depending on the temperature

Density-temperature change graphic is shown in Figure 2. The pre-sintering raw density of the plated sample was estimated to be 1,82gr/cm^3. The highest post-sintering density was achieved at 600°C as 2,03gr/cm^3.

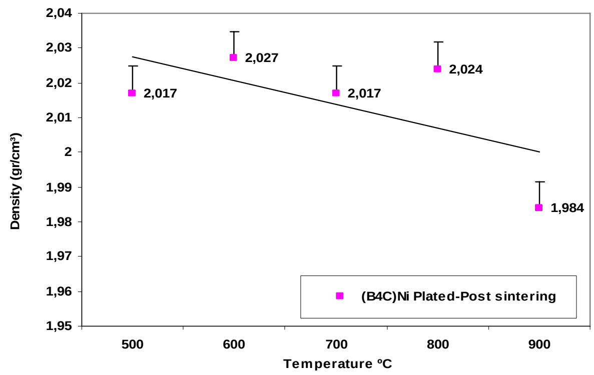


Figure 2. The graphic of density change depending on the temperature

3.2. Analysis of the Mechanical Properties of the Samples

Compression strength and microhardness of the prepared ceramic-metal composite material was mechanically tested. The relation between the sintering temperatures and compression strength values is shown in Figure 3.

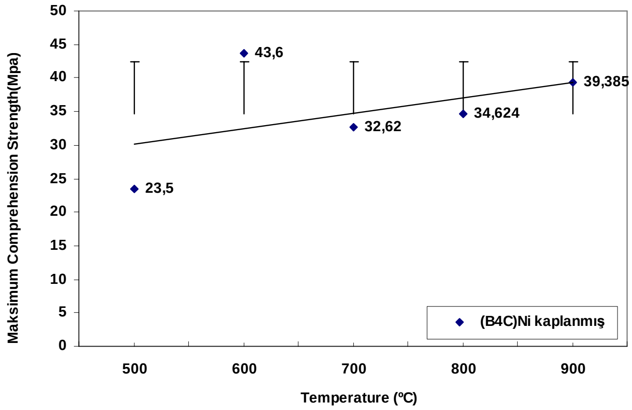


Figure 3. Compression strength test applied to the samples sintered at different temperatures

The microhardness-temperature change graphic is shown in Figure 4. The microhardness values of the composite samples produced using microwave sintering technique within the temperature range 500-900°C from powders obtained as a result of plating B_4C powders through electroless Ni plating method in microwave furnace were given. According to this, the highest microhardness value in the composite samples produced using electroless plating method was observed to be 53,35HV at 800°C

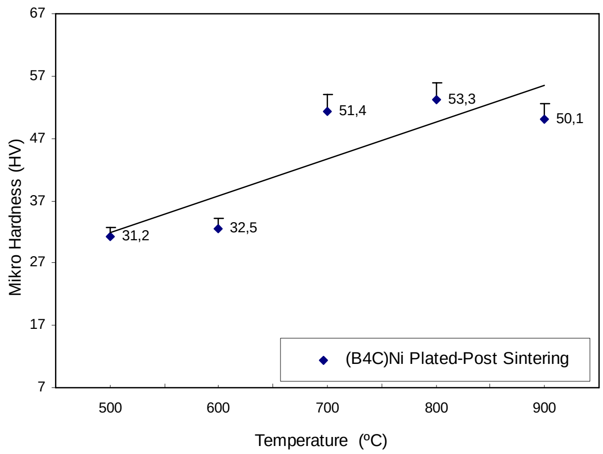
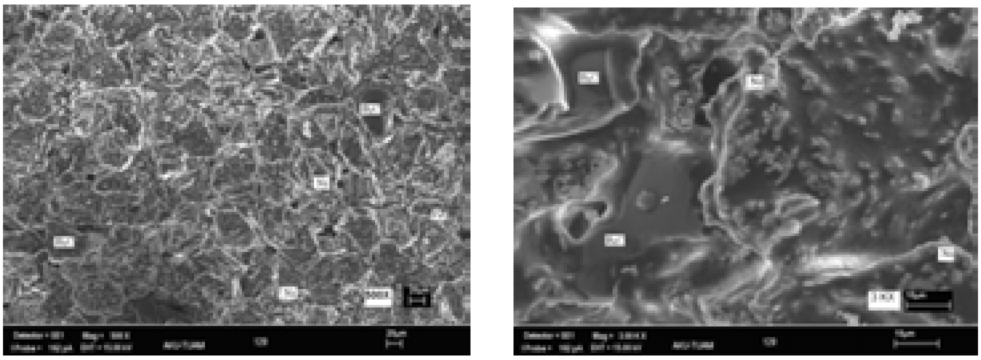


Figure 4. Microhardness test applied on the samples sintered at different temperatures

3.3. Metallographic Analysis

After securing the samples sintered at different temperatures within resin and completing their surface polishing processes, their photographs were taken using scanning electron microscope with magnification of 500X-10kX. After nickel plating process, whether the plating was achieved in B_4C powders or not was examined through SEM analysis. The SEM pictures with different magnification of the ceramic-metal composite sample obtained from (B_4C) powders plated with Ni and sintered at 600°C are given in Figure 5. In Figures 5 a and b, it can be seen that particles were plated with Ni in (B_4C) Ni composite. It was observed that grains were bonded to each other and the particles grew larger. In addition, there were pores exhibiting homogeneous dispersion among the grains.



(a) (b)

Figure 5. SEM view of (B_4C) Ni composite at 600°C

Scanning Electron Microscopy (SEM) and XRD analyses were carried out on the specimens to reveal the effect of Ni plating and characterize the phases present within the specimen. Figure 6 shows Ni plated particles having a layer of Ni with low density of porosity before microwave sintering process. EDX analysis reveals Ni and B presence.

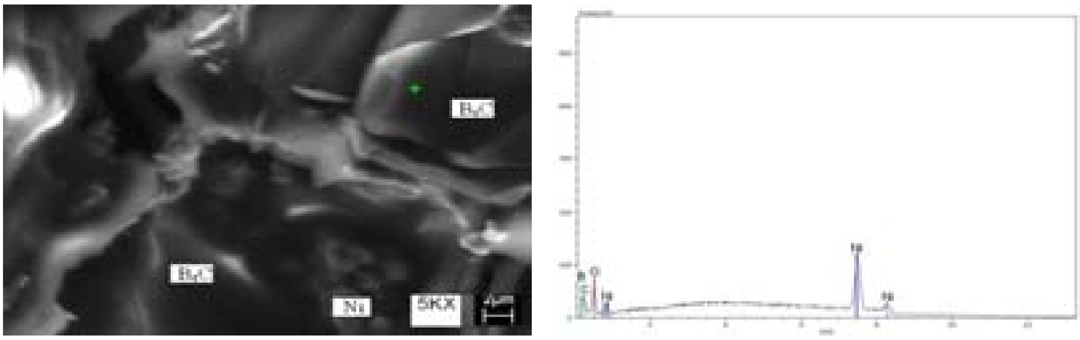


Figure 6. SEM images and EDX analysis from (B_4C)Ni plated specimens

3.4. XRD Analysis

In Figure 7, B_4C and Ni peaks can be seen in the XRD graphic of (B_4C)Ni composite sintered in microwave furnace at 600°C.

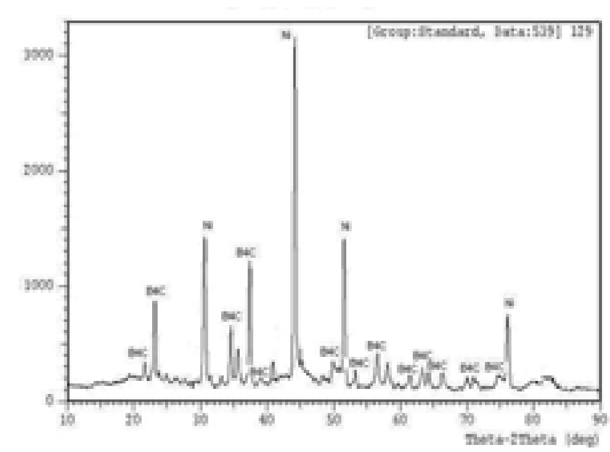


Figure 7. The XRD graphic of Nickel Plated B_4C composite Sintered in Microwave Furnace at 600°C

4. RESULTS AND DISCUSSION

The following results were concluded from the experimental findings:
The highest compression strength was obtained as 43,32MPa at 600°C.
The highest density in composite made from Ni-plated B_4C powder sintered at different temperatures was obtained as 2,03gr/cm^3 at 600°C. The pre-sintering density in the plated sample was estimated to be 1,82gr/cm^3.

- The highest microhardness in composite samples fabricated using electroless Ni-plating method was found as 53,35HV at 800°C.

It was determined that the plated samples have more homogenous microstructures and less pores. It was also found out that the mechanical properties of the plated samples are higher than those of the non-plated samples.

It was concluded that B_4C powders give positive results to Ni-plating and microwave sintering method is more advantageous than classical sintering technique due to its temperature, duration and low energy consumption.

5. ACKNOWLEDGEMENTS

We would like to extend our thanks to the Scientific and Technological Research Council of Turkey (TUBITAK) due to its contributions to this study with project no. 106T744.

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