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Microwave Sintering of Electroless Ni Plated WC Powders

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ABSTRACT

Nickel matrix reinforced with WC has been manufactured by microwave sintering at various temperatures. A uniform nickel layer on WC 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 WC powders to form compounds is controlled through Ni layer existing on the starting powders. A composite consisting of quaternary additions, a ceramic phase, WC, within a matrix of Ni WC and etc., has been prepared at the temperature range 500°C-900°C under Ar shroud. XRD (X-Ray diffraction, SEM (Scanning Electron Microscope), compressive testing and hardness measurements were employed to characterize the properties of the specimens. Experimental results carried out for 900°C suggest that the best properties as σmax and Vikers Hardness (HV) were obtained at 900°C and the microwave sintering of electroless Ni plated WC powders can be used to produce ceramic reinforced Nickel composites.

Key Words: *Microwave sintering, Powder metallurgy, Ceramic-Metal composites and electroless Nickel plating.*

1. INTRODUCTION

Electroless nickel platings have found wide uses in many fields, because the technique of electroless plating was invented and the high performance product with high hardness, wear resistance and corrosion resistance were produced. Several advantages like low cost, easy formation of a continuous and uniform coating on the surface of substrate with complex shape, and capability of depositing on either conductive or nonconductive parts have attracted a lot of interests from the academy and the industry [1]. As compared with other carbides, tungsten carbide (WC) combines favorable properties, such as high hardness, a certain amount of plasticity and good wettability by molten metals [2]. The main disadvantage of WC is its low heat of formation which makes it easily dissolved by molten metals, and the brittleness of WC-base composites (cemented carbides) limits their usefulness in certain wear applications where a combination of high hardness and toughness is needed.

The production of composite materials, including electrochemical coatings, has been of interest up to now. Electrochemical coatings consist of a heterogeneous system of the electrodeposited metal and of various kinds of micrometer-sized particles (oxides, borides, carbides, nitrides, etc.) [3], [7].

The ceramic composites with WC content have higher thermotechnical properties and an increased chemical stability [8]. WC powders interact with the microwave radiation at ambient temperature and the electromagnetic field energy is transformed in heat [9]. In this study, the ceramic-metal composites were obtained by using electroless nickel (Ni) plating with Tungsten carbide (WC) powders.

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2. MATERIAL-METHOD AND PREPARATION OF SAMPLE

The structure and wear properties of new powder metallurgically manufactured nickel-based superalloy matrix composites were investigated in the present study.

2.1. Materials

In this study, Tungsten carbide (WC) was used as ceramic powder and Nickel (Ni) powder as metal. WC powders with 10µm grain size and 99.5% purity and Ni powders with 3µm size and 99.5% purity, which were both provided from Johnson Matthey Materials Technology Company, were used. The aim of this study was to reinforce WC 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 ($NiCl_2.6H_2O$) used in electroless nickel plating bath [10].

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 WC-Ni powders for a day was shaped in hydraulic press coolly under 200 bar and made ready for sintering. In the second method, tungsten 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, 70% WC powders, Nickel chloride, Ammoniac, Hydrazine hydrate and distilled 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	Conditions
Tungsten Carbide (WC)	21 g
Nickel Chloride ($NiCl_2.6H_2O$)	36 g
Hydrazine Hydrate ($N_2H_4.H_2O$)	20%
Distilled Water	80%
Temperature (°C)	95°C
pH Value	10

Nickel plated tungsten 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.

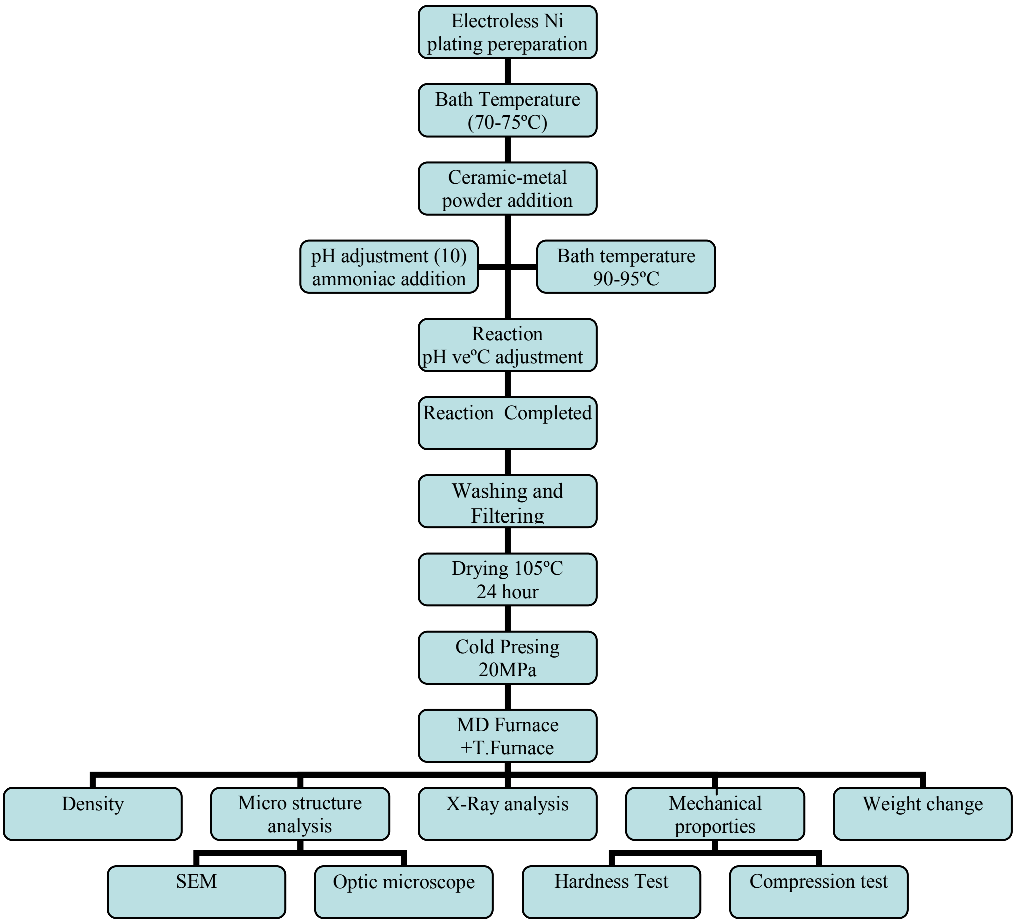


Figure 1. Experiment flowchart.

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 the first method; WC powder used by electroless nickel plated and in the second method; non plated WC powder was used during the sample preparation.

In Figure 2, 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.

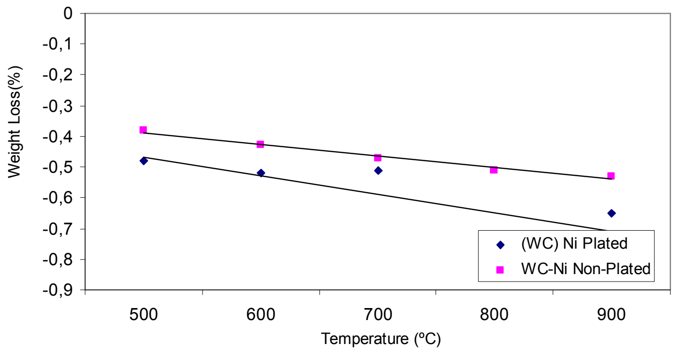


Figure 2. Percent weight change in the sintered samples depending on the temperature.

Density-temperature change graphic is shown in Figure 3 The highest post-sintering density was achieved at 500°C as 7.44gr/cm^3. The pre-sintering density of the non-plated density was estimated to be 6.52 gr/cm^3. The highest post-sintering density was observed to be 7.45gr/cm^3 at 800°C.

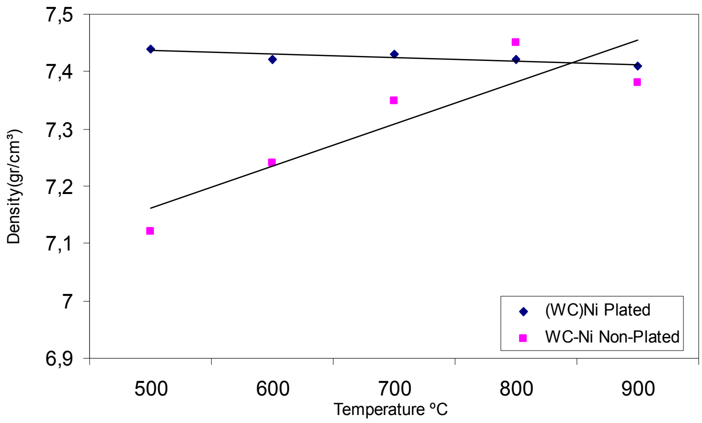


Figure 3. The graphic of density change depending on the temperature.

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 5. The highest plated sample post-sintering comprehension was achieved at 900°C as 27.02MPa. The highest non-plated sample post-sintering comprehension was achieved at 900°C as 17.25MPa.

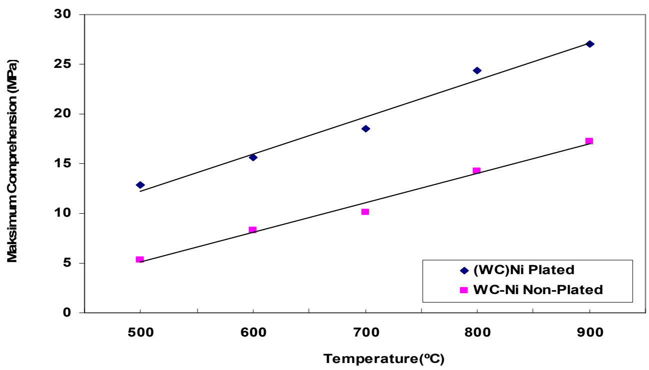


Figure 5. Compression strength test applied to the samples sintered at different temperatures.

The microhardness-temperature change graphic is shown in Figure 6. 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 WC powders through electroless Ni plating method and the samples produced by sintering WC-Ni powders at the same temperatures 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 452HV at 900°C while the highest microhardness value in the samples produced with non-plated powders was observed to be 224HV at 900°C. The microhardness of the plated sample was higher than that of the non-plated sample

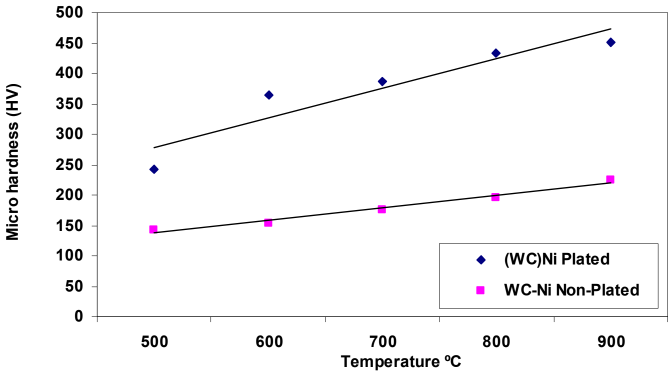
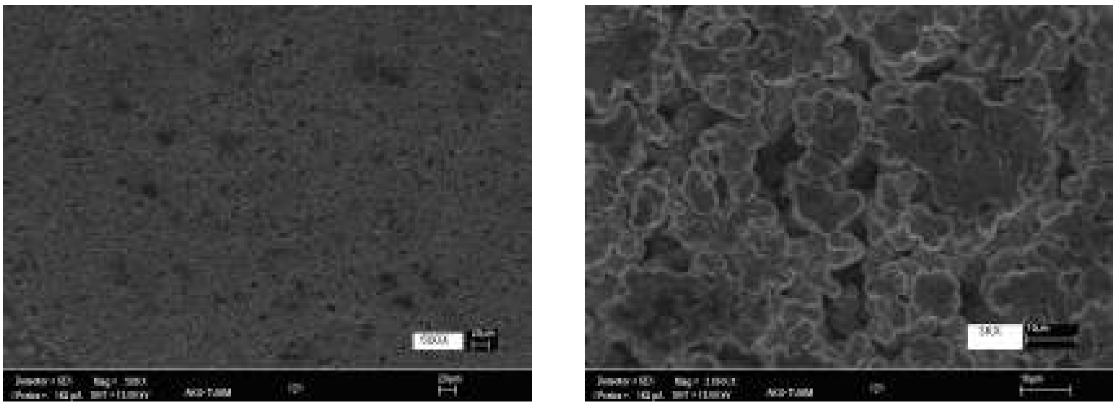


Figure 6. Microhardness test applied on the samples sintered at different temperatures.

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.

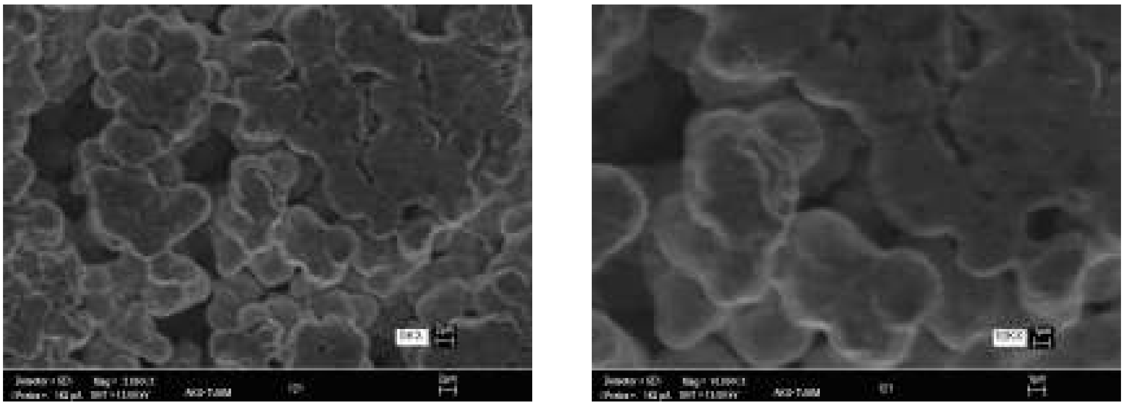
After nickel plating process, whether the plating was achieved in WC powders or not was examined through SEM analysis. The SEM pictures with different magnification of the ceramic-metal composite sample obtained from (WC) powders plated with Ni and sintered at 800°C are given in Figure 7. In Figures 7 a and b, it can be seen that particles were plated with Ni in (WC)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 7. SEM view of (WC)Ni composite at 900°C.

The SEM pictures of ceramic-metal composite sample obtained from plated (WC)Ni powders sintered at 900°C are given in Figure 8a-b. In Figure 8b, WC particle with magnification of 10kX can be seen clearly.



(a) (b)

Figure 8. SEM view of (WC)Ni composite at 900°C.

3.4. XRD Analysis

In Figure 9, WC and Ni peaks can be seen in the XRD graphic of (WC)Ni composite sintered in microwave furnace at 900°C.

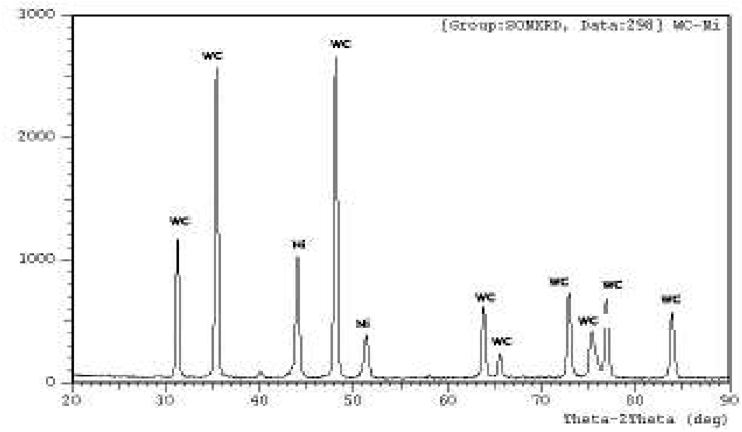


Figure 9. The XRD graphic of Nickel Plated WC composite Sintered in Microwave Furnace at 900°C.

4. RESULTS AND DISCUSSION

The following results were concluded from the experimental findings:

- The highest compression strength was obtained as 27.02MPa at 900°C (Figure 5).

  The highest non-plated sample post-sintering comprehension was achieved at 900°C as 17.25MPa.

- The highest density in composite made from Ni-plated WC powder sintered at different temperatures was obtained as 500°C as 7.44gr/cm^3 (See Figure 3).

- The pre-sintering density in the non-plated sample was estimated to be 6.52 gr/cm^3. The highest post-sintering density was found as 7.45gr/cm^3 at 800°C.

- The highest microhardness in composite samples fabricated using electroless Ni-plating method was found as 452HV at 900°C. The highest microhardness value in the samples fabricated with non-plated powders was tested to be 224HV at 900°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 WC 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.

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