Usage of Electroless Ni Plated WC-Ti Powders in Ceramic-Metal Composite

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Abstract

Metal matrix composites are employed in manufacturing applications such as cutting disks and tips. Ni-Ti metal matrix composites, that is used as an aerospace alloy, have been fabricated by tube furnace sintering at various temperatures with a reinforcing agent of WC. A mixture of %50Ni, %46.66WC and %3.33Ti powders was sintered in a tube furnace under Ar shroud and electroless plating technique was employed to deposit a uniform layer of Ni. One of the benefits that electroless plating provides is that it allows increased density by closer surface contact. A temperature range of 1000-1400C was used to sinter powders, which resulted in a composite consisting of a ceramic phase, WC in a metal matrix of WC Ni, NiTi. Several characterization techniques have been used to reveal the mechanical and physical properties of specimens, such as X-Ray diffraction, SEM (Scanning Electron Microscope), density and hardness measurements. Experimental results suggest that the highest sintering temperature resulted in a hardness of 429.1HV at 1400C. Electroless plating of %50Ni, %46.66WC and 3.33Ti powders provided a better bonding than conventional techniques and is a promising technique to produce ceramic reinforced NiWC Ti composites.

Keywords

Ceramic-metal composites, Electroless nickel coatings, Powder metallurgy

1. INTRODUCTION

Composite materials containing a heterogeneous system with an electrodeposited metal and micrometer-sized particles have ever been of great interest to engineers and scientists and nowadays are still high topic for many applications [1-7]. WC and SiC are known as hard materials are used in cutting tools and because of their splendid hardness, reasonable fracture toughness and high wear resistance.

Conventional sintering methods such as inert gas protected tube furnace are known as effective to produce fully dense parts and used to make ceramic-metal composites [8]. Composites of titanium carbide and iron powders plated with nickel have been widely used as cermet materials to produce various dies and tool parts [9-11]. Ceramic-metal powders can be easily sintered and ceramic-metal composites with superior quality are produced by mixing size controlled elemental particles that are more uniformly distributed. Many studies have focused on the production of high strength cermet materials such as developing direct fabrication methods for a uniformly distributed fine composite powder [12,13]. Variety of applications need materials made of carbides of tungsten, boron, silicon, titanium and tantalum for many years. Generally, these materials are very hard and possesses superior wear resistance as well as oxidation resistance [14]. Electroless nickel plating technique is widely used in many fields, therefore, it allows a high performance product with high density, wear and corrosion resistance. This method has a number of advantages, i.e. low cost and easy formation of a continuous and uniform coating on the surface of substrate with complex shape. Engineers and scientists are very much interested in the capability of depositing a layer of metallic, non metalic or hard coatings on either conductive or nonconductive parts [15]. Metallic materials with small grains exhibiting a high strength are in high interest from both theoretical and experimental point of view. Further enhancement of their mechanical properties is possible due to reinforcement by ceramic particles [16-19].
In this research, the ceramic-metal composites were obtained by using electroless nickel (Ni) plating of WC and Ti powders and in order to produce dense compacts which were followed by compaction and sintering of powders in conventional tube furnace. In order to characterize microstructures and phases formed during the sintering process, X-ray and SEM techniques were employed following the production of specimens.

2. EXPERIMENTAL DETAILS

The idea of this study was to reinforce WC ceramic powders with Ni-Ti. In this research there was used Tungsten Carbide (WC) (size -10µm, purity - 99.5%) as a ceramic powder and Titanium (Ti) (size -40 µm, purity - 99.5%) as metal powder. Both powders were got from Sigma Aldrich Company (Steinheim, Germany). It was thought that Ni powders can either be added in the mixture directly or obtained through plating with nickel chloride (NiCl2.6H2O) used in electroless nickel plating bath. After nickel plated WC-Ti powders for making ceramic-metal composites. It was thought that Ni powders obtained through plating with nickel chloride (NiCl2.6H2O) used in electroless nickel plating bath [7].

2.1. Materials and Procedures

The samples for the experiment were prepared through electroless nickel plating methods. The electroless nickel plating technique was used for the both kinds of the powders and then they were shaped in hydraulic press again coolly under 300 bar pressure. The the shaped samples were sintered for 2 hours within the temperature range 1000-1400°C under argon gas atmosphere in tube furnace. So, the samples were ready for mechanical and metallographic analyses. In electroless Ni plating baths there are used % 46.66WC and %3.33Ti powders, Nickel chloride, Ammoniac, Hydrazine hydrate and distilled water. The contents of the plating baths can be seen in Table 1 and Table 2.

**Table 1. Chemicals of Ni plating bath-1 and their ratios**

<table>
<thead>
<tr>
<th>Chemicals</th>
<th>Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tungsten Carbide(WC)</td>
<td>14g</td>
</tr>
<tr>
<td>Nickel Chloride ( NiCl2.6H2O)</td>
<td>28g</td>
</tr>
<tr>
<td>Hydrazine Hydrate (N2H4.H2O)</td>
<td>20%</td>
</tr>
<tr>
<td>Distilled Water</td>
<td>80%</td>
</tr>
<tr>
<td>Temperature(°C)</td>
<td>90-95 C</td>
</tr>
<tr>
<td>pH Value</td>
<td>9-10</td>
</tr>
</tbody>
</table>

**Table 2. Chemicals of Ni plating bath-2 and their ratios**

<table>
<thead>
<tr>
<th>Chemicals</th>
<th>Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium (Ti)</td>
<td>1g</td>
</tr>
<tr>
<td>Nickel Chloride ( NiCl2.6H2O)</td>
<td>32g</td>
</tr>
<tr>
<td>Hydrazine Hydrate (N2H4.H2O)</td>
<td>20%</td>
</tr>
<tr>
<td>Distil Water</td>
<td>80%</td>
</tr>
<tr>
<td>Temperature(°C)</td>
<td>90-95 C</td>
</tr>
<tr>
<td>pH Value</td>
<td>9-10</td>
</tr>
</tbody>
</table>

The sintered samples were SEM-EDX analyzed and sintering was performed at the temperature range of 1000-1400°C in tube furnace. The device for taking SEM photographs was LEO143OVP Röntech device. Furthermore, in order to measure the compression strength of the samples it was used Shimadzu-AG/IS 100kN testing device and the micro hardnesses of the samples were measured with Shimadzu HMV 2L micro hardness device. The micro hardness measures were obtained by taking the mean of micro hardness values.
In order to calculate the volumetric changes of %50Ni, %46.66WC and %3.33Ti composite material after sintering it was used \( (d=m/V) \) formula (Fig. 1). And Archimedes principle was adopted to measure the volume of post-sintered samples. All the percentages and ratios are given in weight percent unless stated otherwise.

### 2.2. Characterization of Specimens

In the study, the prepared and shaped samples were sintered at temperatures ranging from 1000°C to 1400°C in conventional furnace and made ready for physical, mechanical and metallographic analyses. Density-temperature change curve is shown in Figure 1. The highest sintered density was achieved at 1400°C as 8.47/cm³.

![Figure 1. The density tests results from sintered specimens treated at different temperatures](image1)

The diagram on Fig. 2 shows the micro hardness-temperature change. The micro hardness values of the composite samples were produced using conventional sintering technique within the temperature range 1000-1400°C. According to this, the highest micro hardness value in the composite samples produced using powder metallurgy method was observed to be 429.1HV at 1400°C.

![Figure 2. The micro hardness tests results from sintered specimens treated at different temperatures](image2)
After nickel plating process SEM analysis was made in order to examine if the plating was achieved in \%50Ni,\%46.66WC and \%3.33Ti powders or not. It was observed that grains were bonded to each other and the particles grew larger. In addition, among the grains there were pores exhibiting homogeneous dispersion.

a) 1000°C 1kX NiWCTi composite

b) 1000°C 2kX NiWCTi composite
There were carried out Scanning Electron Microscopy (SEM) and XRD analyses on the specimens to reveal the effect of Ni plating and characterize the phases present within the specimen. Fig. 4, shows Ni plated particles having a layer of Ni with low density of porosity before sintering process.

**Figure 3. The SEM results from sintered NiWCTi composites treated at 1000°C temperatures**

1000°C 3kX NiWCTi composite

1400°C 1kX NiWCTi composite
b) 1400°C 2kX NiWCTi composite

c) 1400°C 3kX NiWCTi composite

**Figure 4.** The SEM results from sintered NiWCTi composites treated at 1400°C temperatures
Figure 5. The EDX results from sintered NiWCTi composites treated at 1000°C temperatures
There were carried out X-Ray diffraction XRD analyses on the specimens to reveal the effect of Ni plating and characterize the phases present within the specimen. Fig. 7 shows it will decide to X-ray analysis taken sample from sintered at 1000 °C According to the X-ray analysis some pics are determined belong to Ni, Ti, NiTi,Ni,Ti and WC.

It can be seen on Fig. 7 that WC reinforced to NiTi and Ni,Ti metallic phases.

**Figure 6.** The EDX results from sintered NiWCTi composites treated at 1400°C temperatures
Moreover, (\%46.66\text{WC} and \%3.33\text{Ti}) powders were plated 10\% Ni and then sintered in a conventional furnace within the temperature range 1000°C, 1100°C, 1200°C, 1300°C, and 1400 °C. After sintering, it was observed a considerable drop in the mechanical properties of specimens. It means that (\%46.66\text{WC} and \%3.33\text{Ti}) 50\% Ni particles were occured by NiTi, Ti, Ni, Ni_Ti and WC phases at 1400 °C and Hardness test results suggest that (\%46.66\text{WC} and \%3.33\text{Ti}) \%50Ni composite sintered at 1400°C shows hardness values respectively.

Figure 7. The XRD results from sintered NiWCTi composites treated at 1000°C temperatures

Figure 8. The XRD results from sintered NiWCTi composites treated at 1400°C temperatures
3. CONCLUSIONS

The following results were concluded from the experimental findings

• The highest density in composite made from %50Ni,%46.66WC and %3.33Ti powders sintered at different temperatures was obtained as 1400°C. The highest density sample was found as 8.47 gr/cm³ at 1400°C.

• The highest hardness %50Ni,%46.66WC and %3.33Ti composite samples fabricated using powder metallurgy method was found as 429.1 HV at 1400°C.

• It was found out that the composition %50Ni,%46.66WC and %3.33Ti at 1400°C has the best properties.
• Wetting problems have been observed on the sample produced by the nickel plating without electric current, and it is considered that the Ni-Ti reaction is realized by using the NiTi equilibrium diagram.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

REFERENCES


