

FABRICATION OF Ni METAL MATRIX COMPOSITES REINFORCED WITH SiO₂ BY MICROWAVE SINTERING

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Abstract

Nickel matrix reinforced with SiO₂ has been manufactured by microwave sintering at various temperatures. A uniform nickel layer on SiO₂ 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 SiO₂ powders to form compounds is controlled through Ni layer existing on the starting powders. A composite consisting of ternary additions, a ceramic phase, SiO₂, and a metal, Ni has been prepared at the temperature range 700°C-1100°C under Ar shroud. XRD, SEM (Scanning Electron Microscope), compressive testing and hardness measurements were employed to characterize the properties of the specimens. Experimental results carried out for 1100°C shows the best properties as σ_{max} and hardness (HB) were obtained at 1100°C. XRD studies revealed that NiO, SiO₂ and phases were formed between SiO₂ and Ni layers, suggesting that microwave sintering of electroless Ni plated SiO₂ powders can be used to produce ceramic reinforced Nickel composites.

Keywords: Microwave sintering, Powder metallurgy, Ceramic-Metal composites and Electroless nickel plating

1. Introduction

Ceramic matrix composites (CMCs) combine reinforcing ceramic phases with a ceramic matrix to create materials with new and superior properties. In ceramic matrix composites, the primary goal of the ceramic reinforcement is to provide toughness to an otherwise brittle ceramic matrix. Fillers can also be added to the ceramic matrix during processing to enhance characteristics such as electrical conductivity, thermal conductivity, thermal expansion, and hardness. The desirable characteristics of CMCs include high-temperature stability, high thermalshock resistance, high hardness, high corrosion resistance, light weight, nonmagnetic and nonconductive properties, and versatility in providing unique engineering solutions. The combination of these characteristics makes ceramic matrix composites attractive alternatives to traditional processing industrial materials such as high alloy steels and refractory metals. For the processing industry, related benefits of using ceramic composites include increased energy efficiency, increased productivity, and regulatory compliance. Key barriers to the broad application of ceramic matrix composites include the lack of specifications, databases, attachment concepts, inservice repair methodology, high cost, and scale-up [1].

Recently, there has been an increasing interest in the synthesis of cerium oxide because it is used as an oxygen conductor in solidoxide fuel cells (SOFC). Cerium oxide

has found applications such as catalysis, ionic conduction, optical additive, etc. [2]. Optical properties of oxide materials based on silica and ceria have attracted much attention. For example, cerium oxide doped silica materials are intensively investigated from the viewpoint of their potential applications as stable luminescent materials for phosphors, scintillators and detector [3]. Optical absorption properties of silicacoated CeO₂ have been reported recently [4,5]. In the case of the core-shell type particles, however, the optical absorbance is dominated by the cerium oxide core and the position of the absorption edge is fundamentally untunable. The synthesis of nanoparticles and control of their properties are important in many critical areas of modern technology such as catalysis, ceramic processing, solar energy conversion, pharmaceuticals, and photography. The effect of size on the electronic and optical properties of the nanosize particles is an area of fundamental interest during the growth of the crystallite from the molecular level to the bulk material [6]. Many approaches have been explored for the preparation of spherical ultrafine particles, including the use of colloids, polymers, glasses, and micelles to successfully control aggregation [7–9]. Many new and unusual physical and chemical properties also arise as particles attain nanosize dimension [10,11]. There is increasing recognition that aqueous synthesis offers growth control capabilities that can be conveniently exploited in preparing these desirable fine particles [12]. Compared to conventional solid-state reaction methods, solution-based synthesis results in higher levels of chemical homogeneity. Also, in solution system, mixing of the starting materials is achieved at the molecular level, and this is especially important when multi-component oxides are being prepared. In addition, surface coating or surface modification of a nanometer semiconductor and metal particles offers a new challenge to synthesis. Among the various chemical synthesis methods [13], the object of this study is to prepare CeO₂-doped SiO₂ nanoparticles by a combined reverse micelle and sol-gel process.

In this study, the production of a tracing pin for soil processing industry is aimed by using ceramic-metal composites. From the previous studies, it is known that the composites was carbide based ceramics while in this study the matrix is oxide based ceramics due to the reason that the cost of oxide ceramics is cheaper than that of carbide ceramics.

2. Experimental Procedure

In this study, specimens were prepared using two different techniques. Initially, Silicon oxide (SiO₂), nickel (Ni) powders at certain rates were mixed homogenously. As a second preparation technique, Silicon oxide, and plated with electroless nickel. Nickel chloride (NiCl₂.6H₂O) was

used for plating. Chemicals used in nickel plating bath and their ratios are shown in Table 1. Ceramic-metal composite was fabricated by microwave sintering specimens prepared in different ways. Sintering was performed at the temperature range 700°C-1100°C under argon atmosphere in microwave oven for two hours.

Table 1. Chemicals of nickel plating bath and their ratios

Chemicals	Conditions
Silicon oxide (SiO ₂)	6g
Nickel Chloride (NiCl ₂ .6H ₂ O)	96g
Hydrazine Hydrate (N ₂ H ₄ .H ₂ O)	20%
Pure Water	80%
Temperature(°C)	95 °C
pH Value	10

3. Experimental Findings

The properties of the composite as sintered specimens were characterized by the physical, mechanical and metallographic examination.

3.1 Examining the Physical Properties of the Composite as Sintered Specimens

The densities of the specimens after sintering were determined (See Figure 1). It can be observed that the density after sintering changed depending on the temperature. The highest density of 3,88 gr/cm³ in plated powders was reached at 1100 °C. The highest density obtained in non-plated specimen was 3,82 gr/cm³ at 1100°C and it was observed that high density can be achieved at low temperature

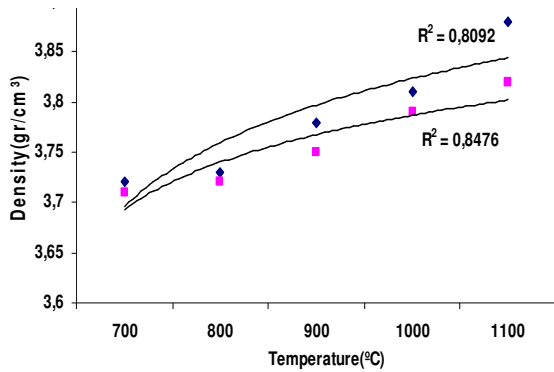


Figure 1. Density-temperature changes in Ni plated specimen

3.2 Weight change

While it was observed that there was a drop in the weights of the specimens sintered after plating, an increase was observed in the weights of the specimens sintered without plating (See Figure 2).

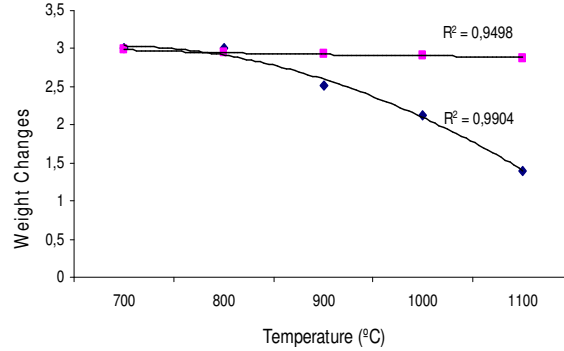


Figure 2. Weight Change (%)

3.2 Mechanical Tests Applied on the Specimens

Compression strength from mechanical tests applied on the specimens after sintering was examined. In Figure 3, compression test was applied on the plated specimens and specimens fabricated without plating. As can be understood from the graphic, the highest strength was achieved as 182MPa at 1100°C in the plated specimen. The highest compression strength in the non-plated specimen was 87,8MPa at 1100°C.

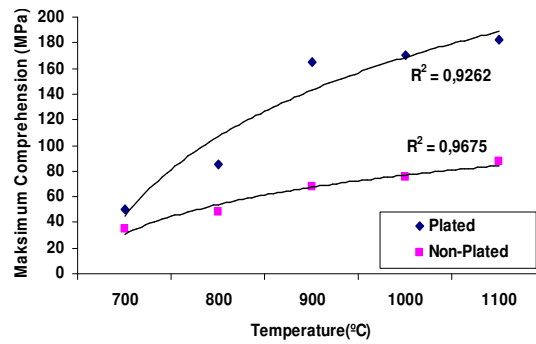


Figure 3. Compression strength – temperature graphic

The Hardness (HB) of the sintered specimens was also measured as well as compression strength. According to the hardness-temperature graphic given in Figure 4 for the sintered specimens, the highest hardness for the plated specimen was obtained as 40,56HB at 1100 °C. The highest hardness for non-plated specimen was obtained as 33,4HB at 1100°C.

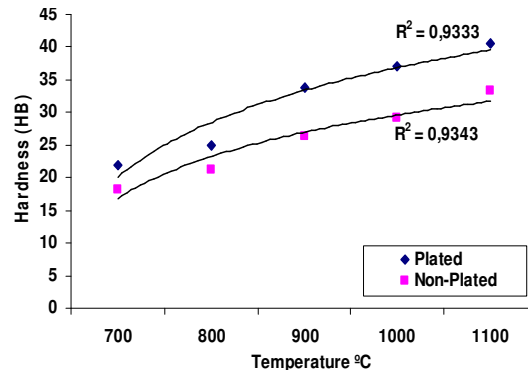


Figure 4. Hardness – temperature graphic

3.3 Metallographic Analysis

After the specimens sintered at different sintering temperatures were secured within resin and surface polishing processes were completed, their photographs were taken and EDX analyses were carried out by SEM (LEO 1430 VP equipped with RONTEC EDX) with magnifications of 500-1kX-2kX-3kX. The SEM photograph of Ni plated SiO₂ powders, which yielded the best result in terms of mechanical strength in the sintered specimens (plated with Ni and prepared by normal mixture method) during the study, after sintering at 1100°C is shown in Figures 5 it can be seen that the structure includes two types of pores, small and large. The pores do not show a homogenous distribution.

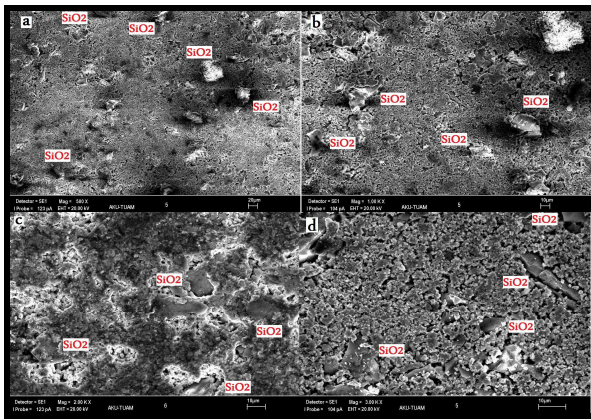
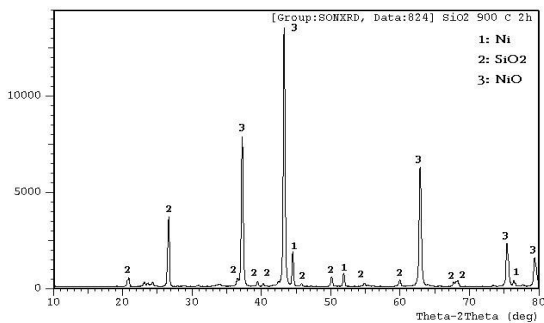


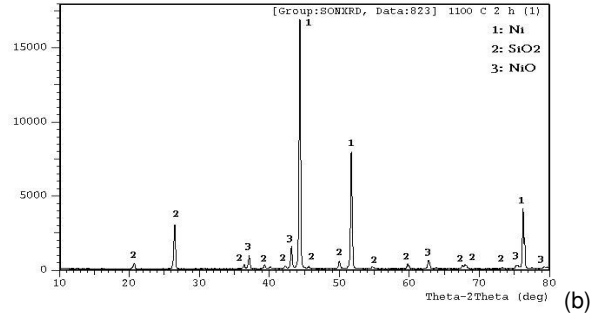
Figure 5. SEM view of (SiO₂)Ni composite at 1100°C

3.4 XRD Analysis

Ni plated powder specimens were characterized by XRD analysis. The XRD analysis of the powder obtained from SiO₂ powders following Ni plating is given in Figure 6a. As can be understood from the analysis results, the existence of Ni peak in the graphs shows that SiO₂ powders were plated with Ni. The XRD analysis of SiO₂ +Ni composite fabricated at 900 °C from powders Ni plated through the same way is shown in Figure 6b. As can be seen, NiO phase occurred in the fabricated ceramic-metal composite.



(a) Ni plated (SiO₂) Composite sintered at 900°C



Plated SiO₂-Ni Composite

Figure 6. Ni plated (SiO₂) Composite sintered at 1100°C

4. Results and Discussion

The following results were concluded from the experimental findings:

- The highest compression strength was obtained as 182MPa at 1100°C (Figure 3).
- The highest density in composite made from Ni-plated SiO₂ powder sintered at different temperatures was obtained as 1100°C as 3,88g/cm³. (Figure 1).
- The highest microhardness in composite samples fabricated using electroless Ni-plating method was found as 40,56HB at 1100°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 SiO₂ 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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