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Fabrication of electroless Ni plated Fe-TiC metal matrix composites

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Abstract

Ni-Fe metal matrix composites reinforced with TiC have been fabricated by tube furnace sintering at various temperatures. A uniform nickel layer on TiC and Fe powders was deposited using electroless plating technique prior to sintering, allowing closer surface contact than can be achieved using conventional methods, such as mechanical alloying. The reactivity between TiC and Fe powders, to form carbides of Fe, is controlled through Ni layer existing on the starting powders. A composite consisting of quaternary additions, a ceramic phase, TiC, within a matrix of FeNi, has been prepared at the temperature range 1000–1400°C under argon shroud. X-ray diffraction, scanning electron microscope (SEM), compressive testing and hardness measurements were employed to characterize the properties of the specimens. Experimental results carried out at 1200°C suggest that the best properties, such as maximum compression strength (σ_{max}) and hardness (HB), were obtained at 1200°C and the tube sintering of electroless Ni plated TiC and Fe powders, is a promising technique to produce ceramic reinforced Ni-Fe composites.

Keywords: composites; electroless nickel plating; powder metallurgy; sintering.

1. Introduction

The production of composite materials, including composite electrochemical coatings of a heterogeneous system of the electrodeposited metal and micrometer-sized particles, has been of interest up to now [1–7]. Hard materials, such as WC–Ni (tungsten carbide-nickel) and TiC–Ni, are mainly used as cutting tools and in wear resistant applications, because of their superior hardness and reasonable fracture toughness. Generally, ceramic-metal composites are produced by conventional sintering methods, which are productive and produce fully dense parts [8]. Composites of TiC and iron plated with nickel have been widely used as cermet materials for the production of various dies and tool parts [9–11]. Ceramic-metal powders can be easily achieved using elemental particles that are finer and more uniform. There have been many studies on the production of high strength cermet materials, which have been focused on developing direct fabrication methods for a uniformly distributed fine composite powder [12, 13]. The carbides of tungsten, boron, silicon, titanium and tantalum have been used for a variety of applications over the years. In general, they are very hard and possess excellent wear resistance, as well as oxidation resistance [14]. Electroless nickel plating technique, widely used in many fields, therefore, allows a high performance product with high hardness, wear resistance and corrosion resistance. There are several advantages of this method, such as low cost and easy formation of a continuous and uniform coating on the surface of a substrate with complex shape. The capability of depositing on either conductive or nonconductive parts has also attracted much interest from the academy and the industry [15]. Metallic materials with small grains, exhibiting a high strength, are interesting from both a theoretical and an experimental point of view. Further enhancement of their mechanical properties is possible, due to reinforcement by ceramic particles [16, 17].

In this study, the ceramic-metal composites were obtained by using electroless Ni plating of TiC and Fe powders, followed by compaction and sintering of powders in a conventional furnace, to produce dense compacts. X-ray and SEM techniques were employed to characterize microstructures and phases which occurred during the sintering process.

2. Materials and methods

2.1. Materials

In this study, TiC was used as the ceramic powder and Ni and Fe powders as the metal. TiC powders with 10 μm grain size and 99.5% purity and Fe powders with 20 μm size and 99.5% purity, were provided by Sigma Aldrich Company (Steinheim, Germany). The aim of this study was to reinforce TiC ceramic powders with Ni-Fe. It was thought that Ni powders can either be added in the mixture directly or mixture can receive Ni which is extracted during the nickel plating.

2.2. Method and preparation of sample

In the experimental study, the samples were prepared in three different compositions. In the first composition, 50 wt% Fe powders were plated using an electroless nickel plating technique and 10, 20, 30 wt% TiC powders were added before being shaped in a hydraulic press under 200 bar pressure. The shaped samples were sintered for 1 h within the temperature range 700–1100°C under argon gas atmosphere, in a microwave furnace. The sintered samples prepared for mechanical and metallography analysis. In an electroless Ni plating bath, 10%, 20%, 30% TiC powders, nickel chloride, ammoniac, hydrazine hydrate and distilled water by weight were used. The contents of the plating bath are given in Table 1.

Nickel plated TiC-Fe powders were purified from chemicals by washing with distilled water after plating and made ready for subsequent processing.

Sintering was performed at 1000–1400°C in a tube furnace. SEM-EDX analyses were employed on the sintered samples. SEM photographs were taken with an LEO143OVP Röntech device (Carl Zeiss, Germany). Furthermore, a Shimadzu-AG/IS (Shimadzu, Japan) 100 kN testing device was used to measure the compression strength of the samples; the hardness of the samples was measured with a Shimadzu HMV 2L hardness device. The hardness measures were obtained by taking the mean of the hardness values.

3. Results and discussion

3.1. Physical properties

The theoretical densities of Ni plated TiC-Fe powders before and after sintering are given in Figure 1. It can be seen that the density after sintering depends on the sintering temperature and it reached the highest value of 6.92 g/cm^3 at 1400°C. The highest density obtained in a non-plated specimen was 7.36 g/cm^3 at 1000°C; a high density of powders can be obtained at lower sintering temperatures.

The difference in the densities can be attributed to the existence of a deposited layer of Ni prior to sintering. Although the nickel layer acts as a binding layer between the neighboring powders, it also increases the inter-particle distance, resulting in the lower density of powders.

3.2. Weight change

Figure 2 shows the weight variations observed in specimens. While there was a drop in the weights of the specimens sintered after nickel 10% TiC+90% (Fe-Ni) plated specimens, a decrease of a higher degree was observed in the weights of the specimens sintered without nickel plating. Such weight variations can originate from the evaporation of volatile substances entrapped within the compacted powders and can be lost during the sintering. While nickel 10% TiC+90% (Fe-Ni), plated specimens can hold less substance, non nickel plated specimens can hold more substance.

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

Chemicals	Conditons
Titanium carbide (TiC)	3 g
Iron (Fe)	15 g
Nickel chloride ($NiCI_2.6H_2O$)	48 g
Hydrazine hydrate ($N_2H_4.H_2O$)	20%
Distilled water	80%
Temperature (°C)	90–95°C
pH Value	10

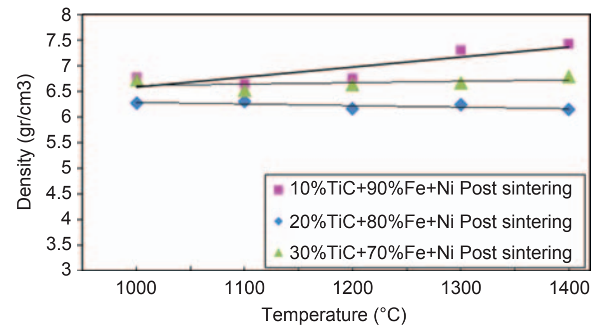

Figure 1 Density-temperature changes in Ni plated specimen.

3.3. Compression strength and hardness

Compression tests and hardness measurements were carried out on all specimens. As can be seen in Figure 3, the highest compression strength was 260 MPa at 1200°C sintering temperature, in Ni plated 10% TiC+90% (Fe-Ni) specimens, whereas the highest compression strength in the 20% TiC+80% (Fe-Ni) specimen was observed to be 172MPa at 1200°C sintering temperature. The highest compression strength in the 30% TiC+70% (Fe-Ni) specimen was observed to be 95 MPa at 1200°C. The compression test results appear to have generated similar results, with a slight variation in values, suggesting that sintering of nickel plated 10% TiC+90% (Fe-Ni) specimens are stronger than the other speimens. Nickel plating yields higher compression values because of the binding nickel layer, which accommodates the compressive forces and allows the powders to yield during testing before failure.

The hardness (HB) of the sintered specimens was also measured. According to the hardness-temperature graphs in Figure 4 for the sintered specimens, the highest hardness values for the 10% TiC+90% (Fe-Ni), 20% TiC+80% (Fe-Ni) and 30% TiC+70% (Fe-Ni) plated specimens, were 94HB, 123HB and 126HB, respectively, at 1200°C.

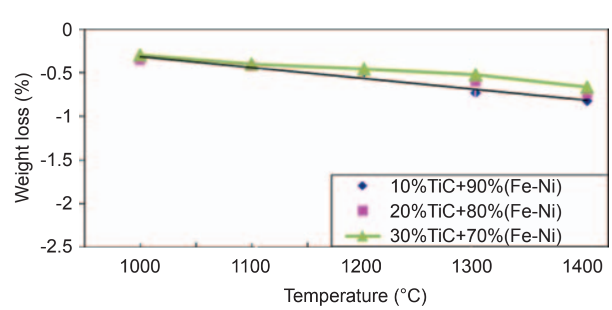

Figure 2 Weight change (%) in both specimen groups.

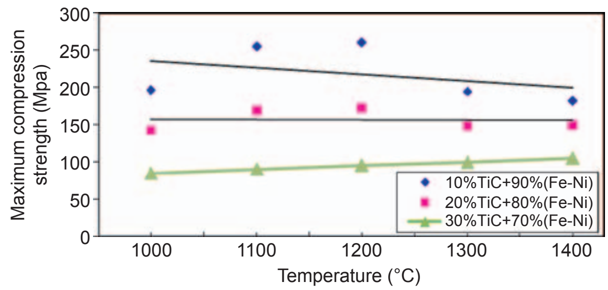

Figure 3 Maximum compression strength (MPa) against sintering temperature.

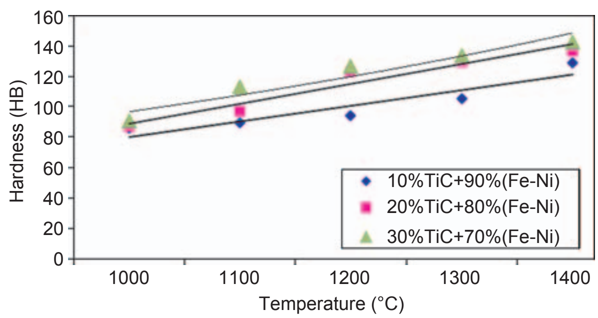

Figure 4 Hardness against sintering temperature.

3.4. Metallographs analysis

After the specimens, sintered at different temperatures, were secured within resin and the surface polishing processes were completed, their photographs were taken and EDX analyses were carried out using SEM (LEO 1430 VP equipped with RONTEC EDX) with magnifications of 500×, 1000×, 2000× and 3000×. The SEM photographs of the Ni plated TiC-Fe powders, which yielded the best results in terms of mechanical strength in the sintered specimens (plated with Ni and prepared by normal mixture method) during the study, after sintering at 1200°C, are shown in Figures 5 and 6. SEM photographs of Ni plated TiC-Fe powders taken after sintering at 1200°C are also given. The SEM analysis results of the ceramic-metal composite specimens obtained from Ni plated (TiC-Fe) powders sintered at 1200°C are shown in Figures 5 and 6. On examination of the SEM photograph in Figure 6A, it can be seen that the structure includes two types of pores; small and large Figure 7. The pores do not show a homogenous distribution.

3.5. XRD analysis

Ni plated powder specimens were characterized by XRD analysis. The XRD analysis of the powder obtained from TiC-Fe powders, following Ni plating, is given in Figure 8A. From the analysis results, the existence of the Ni peak in the graphs shows that TiC-Fe powders were plated with Ni. The XRD analysis of the TiC-Fe-Ni composite fabricated at 1200°C from powders Ni plated in the same way is shown in Figure 8B, which shows the presence of FeNi phase in the fabricated ceramic-metal composite.

4. Conclusion

Following results are obtained from the analysis of results:

- In this study, Iron powders and TiC powders were electroless Ni plated and then sintered in a conventional furnace. After

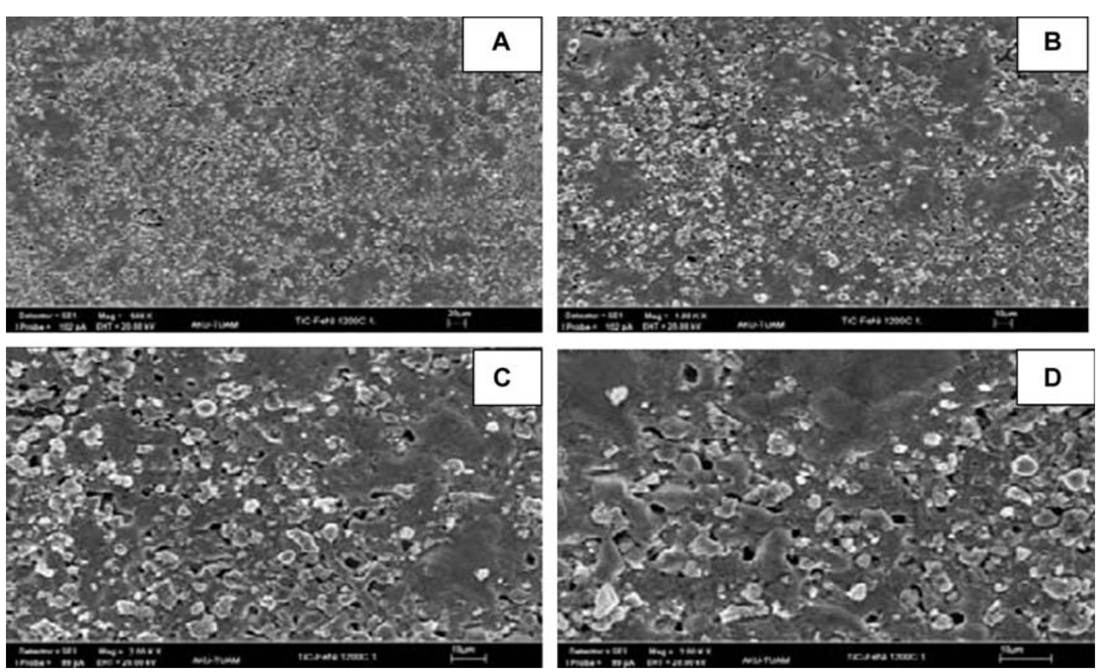

Figure 5 10% TiC+90% (Fe+Ni) 1200°C composite SEM images.

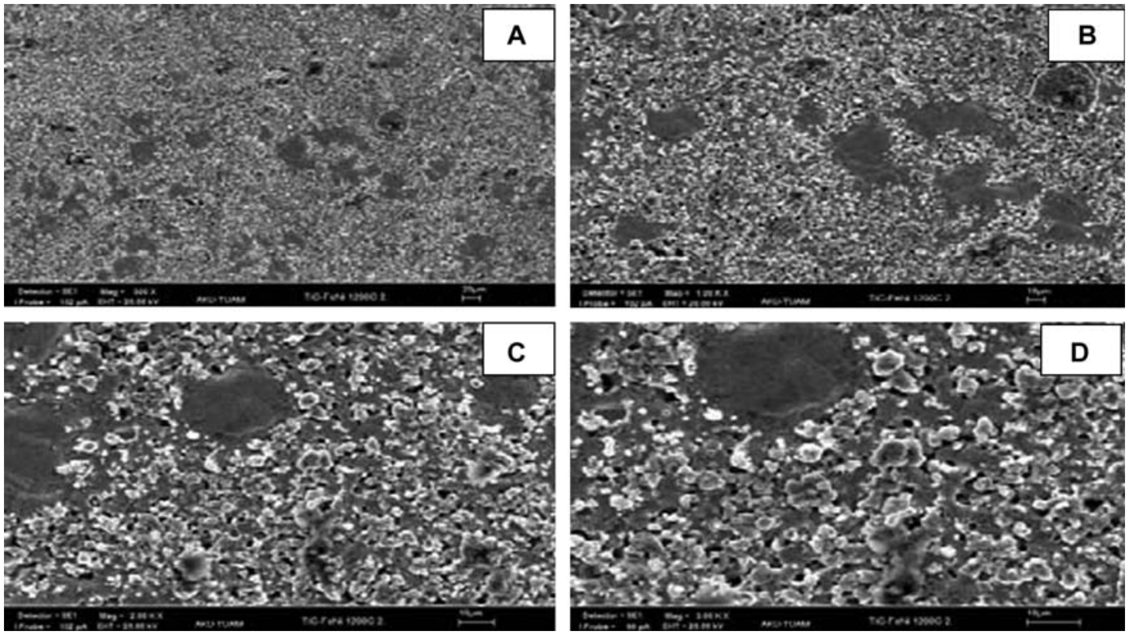

Figure 6 20% TiC+80% (Fe+Ni) 1200°C composite SEM images.

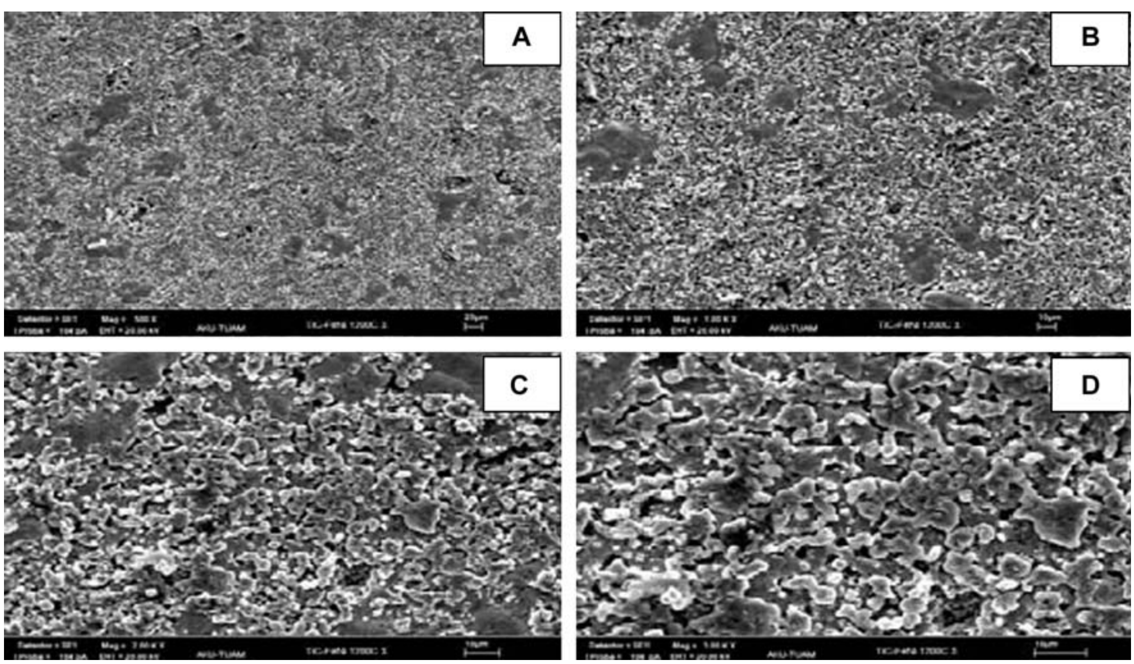

Figure 7 30% TiC+70% (Fe+Ni) 1200°C composite SEM images.

sintering, a considerable drop in the mechanical properties of specimens sintered at 1300°C and 1400°C were observed. It was concluded that TiC particles were not wetted by FeNi intermetallic phase during sintering and the formation of composite structure was therefore believed to be hindered.

- From XRD results of specimens sintered at 1200°C, FeNi intermetallic phase and TiC appears to be coexisting as two separate constituents.
- Hardness test results suggest that 30% TiC+70% FeNi and 10% TiC 90% FeNi mixtures sintered at 1200°C shows Brinell hardness values 126 HB and 90 HB, respectively. It appears that the increase in the amount of TiC is directly related to an increase in hardness values.
- A homogeneous plating onto Fe and TiC powders seems to have been achieved following the metallographic

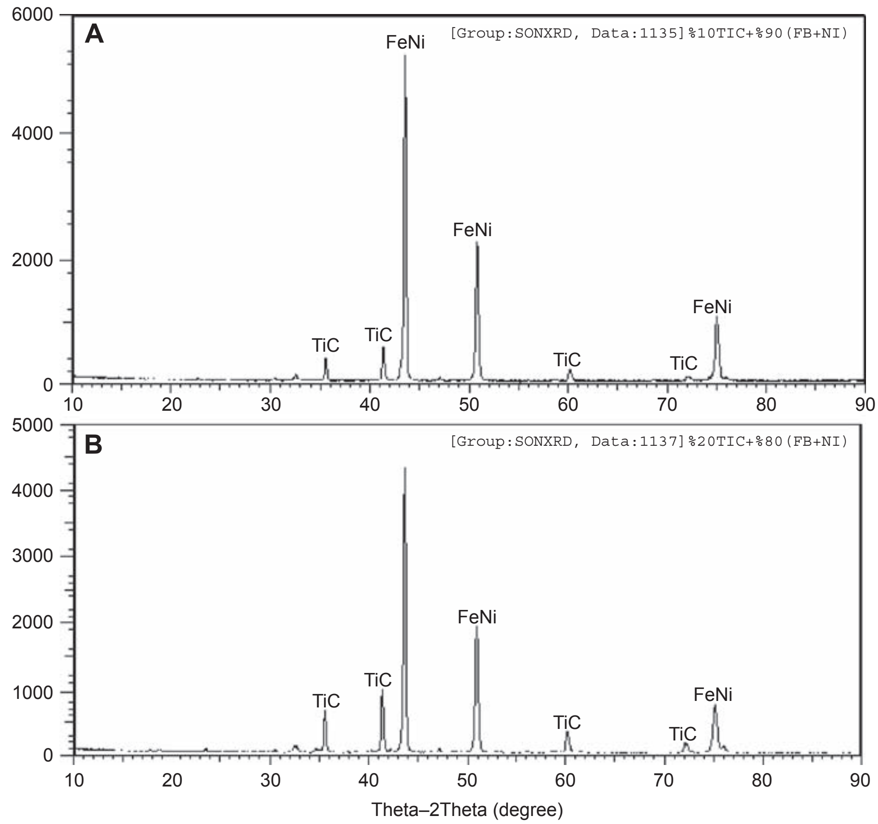

Figure 8 XRD analysis of TiCFeNi composite sintered at 1200°C.

examination. It is also observed that various reactions occurred at the interface of contacting surfaces i.e., between Ni plating and the other constituents i.e., Fe and TiC powders, easing the formation of composite structure.

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