Materials and Design 52 (2013) 572–579

Contents lists available at SciVerse ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Preparation of nickel-coated titanium carbide particulates and their use in the production of reinforced iron matrix composites

Danqing Yi [a,b], Pengchao Yu [a,b], Bin Hu [a], Huiqun Liu [a,b], Bin Wang [a,b,*], Yong Jiang [a,c,*]

[a] School of Materials Science and Engineering, Central South University, Changsha 410083, PR China
[b] Key Lab of Nonferrous Materials, Ministry of Education, Central South University, Changsha 410083, PR China
[c] State Key Laboratory for Powder Metallurgy, Central South University, Changsha 410083, PR China

ARTICLE INFO

Article history:
Received 9 January 2013
Accepted 27 March 2013
Available online 13 June 2013

Keywords:
Electroless plating
Hot isostatic pressing
TiC reinforced iron matrix composites
Microstructure
Mechanical properties

ABSTRACT

Ni-coated titanium carbide (TiC) composite powders were prepared by electroless plating (EP). Further, using hot isostatic pressing (HIP), iron matrix composites reinforced with 4 wt% Ni-coated TiC particulates with relative density close to 100% were prepared. The microstructure and phase composition of the Ni-coated powders and the composites were analyzed using X-ray diffraction, scanning electron microscopy, and energy dispersive X-ray spectroscopy. The results showed that the TiC particles were distributed uniformly in the matrix and were free of segregation or coarsening. Compared to the TiC particles without Ni coating, the reinforced iron-based composites containing the Ni-coated particles showed higher relative densities and better mechanical properties. The density, hardness, tensile strength, and elongation were enhanced to 99.98%, 243 HV, 565 MPa, and 11.7%, respectively in composites containing Ni-coated TiC particles from 99.70%, 210 HV, 514 MPa, and 10.3%, respectively in composites that were prepared using particles without Ni coating. In addition, the mass losses in the composites containing the Ni-coated particles were reduced by 32–75% in the abrasive wear test with various vertical loads. We propose that the nickel coatings on the particulates had a beneficial effect on the microstructure and properties of the reinforced iron-based composites is due to promotion of neck formation and growth between TiC and iron powders during sintering, which enhanced the density of the sintered compact and the bonding strength between the TiC particles and the iron matrix.

1. Introduction

Titanium carbide (TiC) is one of the most suitable reinforcing agents in matrix composites due to its excellent combination of physical properties such as high melting point (3067 °C), low density (4.93 g/cm^3), extreme hardness (2800–3200 HV), high mechanical stiffness, and good thermal and electrical conductivities [1–3]. Therefore, iron-based composites reinforced by TiC particulates have received considerable attention owing to their excellent properties [4,5].

Iron matrix composites reinforced with TiC particulates fabricated by traditional powder metallurgy (PM) have been extensively studied [6,7]. The typical advantages of this route are the economical use of raw materials and low energy costs. In addition, PM allows the mixing of arbitrary proportions of the alloying elements and the addition of ceramic particles. However, the materials produced by this technique generally suffer from contaminated matrix-reinforcement interfaces. In situ synthesis is a recently developed method to fabricate TiC/Fe composites. There are various routes to fabricate metal matrix composites (MMCs) reinforced with TiC particulates prepared in situ such as conventional casting [8–12], self-propagating high-temperature synthesis (SHS) [13–15], and thermite reduction [16]. The reinforced surfaces are likely to be free from gas absorption, oxidation, and other detrimental surface reaction contaminations and the bonding at the interface between the matrix and the reinforcing agent tends to be stronger. However, there are individual disadvantages in each of these methods that are difficult to be avoided. Fe/TiC composites produced by casting confront two challenges. On one hand, the distribution of TiC in the iron matrix is likely to be uneven because of the difference in density between Fe and TiC. On the other hand, the volume fraction of TiC is limited because of reduction of the steel liquid fluidity at high TiC levels. Ren et al. [17] decreased the density difference to a certain degree by substituting TiC by (TiW)C, although the asymmetrical distribution of the hard particles could not be eliminated. The main issue with SHS is that the procedure is only feasible to produce MMCs with low content of the binder phase. According to Zhang et al., the content of nickel

* Corresponding authors. Address: Key Lab of Nonferrous Materials, Ministry of Education, Central South University, Changsha 410083, Hunan, PR China. Tel.: +86 73188830263; fax: +86 73188836320 (B. Wang).
E-mail addresses: wangbin325@263.net (B. Wang), yioffice@csu.edu.cn (Y. Jiang).

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http://dx.doi.org/10.1016/j.matdes.2013.05.097

Table 1
Processing technology of the alloys studied.

Item	Composition	Processing technology
Alloy 1	100 wt% carbonyl iron	HIP
Alloy 2	96 wt% carbonyl iron + 4 wt%TiC	HIP
Alloy 3	96 wt% carbonyl iron + 4 wt%TiC	EP + HIP

Table 2
Composition of the EP bath and operating parameters.

Bath composition		Operating parameters	
Nickel sulfate ($NiSO_4{\cdot}6H_2O$)	70 g/L	Temperature	70 °C
Ethylene Diamine Tetraacetic Acid(EDTA)	25 g/L	pH value	10
Lactic acid	5 vol%	Time	60 min
$N_2H_4{\cdot}H_2O$	10 vol%	Powder load	20 g/L
NaOH	40 g/L		

can't exceed 40 wt% in the in situ synthesis of Ni matrix composites reinforced by TiC by SHS. Further, the relative density of the TiC/Fe composite prepared by SHS remains below 98% [18].

In the present study, a novel process was used to prepare iron-based composite reinforced by TiC particulates. Electroless plating (EP) was used to clad a layer of nickel on the TiC powder to metallize the surfaces of the ceramics. Hot isostatic pressing (HIP) was then employed to obtain the compact iron-based composites reinforced by Ni-coated TiC particulates after mixing the TiC and Fe powders. The microstructure and properties of the composites reinforced with particulates with and without Ni coatings were analyzed and compared, and the effect of surface metallization of the TiC particles on the properties was studied. Effects of reinforcing MMCs with metal-coated ceramics have been investigated by some researchers [19–21]. Usually, matrices of Al, Cu etc., with low melting points have been focused upon and the composites were prepared by immersion, casting, or liquid sintering. Hence, usually, the effects of the wettability of the metal coatings on the ceramics and liquid metal matrix were studied. In the present study, HIP was used to prepare the composite materials, and a molten metal did not emerge since the experimental temperature was below the melting point. As a consequence, the focus is more upon the sintering process to analyze the effect of Ni-coating on the microstructure and properties of the iron-based composites reinforce by TiC. It is expected that the results of this study can be a significant step in promoting the development and applications of iron-based composites reinforced by TiC particulates with excellent performances.

2. Materials and experimental procedures

Commercial TiC powders (4–10 μm, 99.5% purity) and carbonyl iron (Fe) (2–8 μm, 99.5% purity, 0.4% C) were used in the experiments. Three alloys were prepared and the preparation method of each is listed in Table 1. The specific technological process is presented in Fig. 1.

EP was chosen to prepare nickel-coated TiC powders. As phosphorus (P) is detrimental to steel because of its easy segregation on the grain boundaries, in the present study, pure Ni coating was obtained instead of Ni–P coatings, which is commonly used in other investigations. Hydrazine (N_2H_4) was used as the reducing agent. The reaction occurring during the EP is presented in Eq. (1).

$$N_2H_4 + 2Ni^{2+} + 4OH^- \rightarrow N_2 \uparrow + 4H_2O + 2Ni \downarrow \quad (1)$$

EP was carried out in a thermostat water bath with electromagnetic stirring. The chemical composition of the plating solution and the experimental parameters used for the plating are listed in Table 2. The TiC powders were washed ultrasonically sequentially in methanol, diluted sodium hydroxide (NaOH) solution, and hydrochloric acid (HCl) for 10 min to eliminate surface contamination. The powders were then immersed into an aqueous solution of hydrofluoric acid (HF, 50 ml/L), hydrogen nitrate (HNO_3, 80 ml/L), and ammonium fluoride (NH_4F, 2 g/L) for 20 min to coarsen their surfaces after cleaning with deionized water. The etched TiC particles were sensitized in a solution of stannous chloride($SnCl_2$, 10 g/L) and HCl (40 ml/L) and subsequently immersed in an activation solution consisting of stannous chloride ($PdCl_2$, 0.5 g/L) and HCl (10 ml/L). During both procedures, the solutions were stirred strongly at room temperature for 20 min. The plating was carried out after the activated powders had been repeatedly washed in deionized water. After plating for 60 min, the powders were filtered from the plating solution and were washed again with deionized water. The obtained Ni-coated TiC powders were dried in a vacuum drying oven at 80 °C for 12 h.

The TiC powders and carbonyl iron were mixed in a planetary ball mill (XQM-0.4-400, Changsha, China) for 4 h. Alcohol was added as the process control agent. The mixed powders were dried in a hydrogen (H_2) furnace at 500 °C for 2 h and oxides on the surface of the powders were deoxidated. The powders were subsequently subjected to cold isostatic pressing using a latex die at 200 MPa for 15 min to obtain the green compacts. The compacts were pre-sintered for 3 h at 1200 °C in vacuum to obtain samples with relative densities higher than 95%. Then, the sintered compacts were subjected to HIP at 1230 °C and 150 MPa for 4 h.

The relative densities of the sintered specimens were determined by the Archimedes principle. The hardness tests were performed using a Vickers Sclerometer (HV-5). To examine the tensile properties, impact toughness, and wear resistance, the sintered samples were machined into test specimens using electric spark machining. The tensile strength and elongation were measured with a universal tensile tester (CSS-44100, Changchun, China) and the impact toughness was tested using an automatic impact testing machine (CBD-300, Changsha, China). Wear resistance was investigated on an UMT-3 (Ceter, USA) wear testing machine under dry conditions at room temperature. The friction mode reciprocated motion with a total distance of 180 m at a constant sliding speed of 0.1 m/s on a pin-on-disc apparatus, with a chromium steel ball (9.5 mm, HRC62) used as the pin. The tests were carried out at three different vertical loads of 30, 50, and 70 N.

Phase identification of the specimens was carried out on a D/Max 2500 X-ray diffractometer with a graphite monochromator using Cu Kα radiation operated at 30 kV and 40 mA with a scanning speed of 4°/min. For microstructure and morphology analysis, a scanning electron microscope (FE-SEM, SIRION200, FEI, USA) was employed. The microarea elements were analyzed using energy dispersive spectroscopy (EDS). The Ni content in the nickel-coated TiC composite powders was analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES).

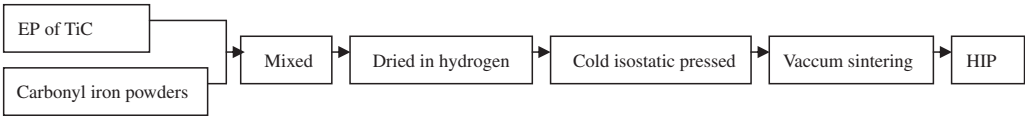

Fig. 1. Schematic diagram of the process.