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Fabrication and mechanical properties of metal matrix composite with homogeneously dispersed ceramic particles

Eun-Hee Kim[a,*], Geun-Ho Cho[a], Je-Hyun Lee[a], Yeon-Gil Jung[a,**], YoungSoo Yoo[b], SeongMoon Seo[b]

[a] *School of Nano and Advanced Materials Engineering, Changwon National University, #9 Sarim-dong, Changwon, Kyungnam 641-773, Republic of Korea*
[b] *High Temperature Materials Research Group, Korea Institute of Materials Science, 797 Changwondaero, Changwon, Kyungnam 641-831, Republic of Korea*

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Abstract

Titanium carbide (TiC) particles were coated with nickel (Ni) to increase their compatibility with a metal matrix, leading to an improvement in the dispersibility of TiC particles in the molten matrix. TiC particles were dispersed into a basic aqueous solution of pH 12, and then nickel nitrate ($Ni(NO_3)_2$), as a Ni precursor, was added to the TiC suspension. The interaction between the TiC particles and the Ni precursor is driven by the attractive force between the Ni cations and the TiC particles with negative charge. An inoculant (ferrosilicon), which has been used in the foundry industry to improve crystal growth of graphite, was used as a core particle. The Ni-treated TiC particles were coated onto the surface of the inoculant using an inorganic binder converted into its glass phase by sol–gel reactions. The reinforcement particles prepared through the dual-coating process were then injected into the molten matrix based on iron at 1500 °C. The crystal phase of the graphite is more finely and shortly grown in the reinforced metal matrix than in that without the reinforcement particles. This means that the reinforcement particles are homogeneously and uniformly dispersed into the matrix without any aggregation of particles, implying that the mechanical properties of the reinforced matrix would be greater than those of a non-reinforced matrix. Consequently, metal matrix composites with reasonable properties can be fabricated successfully using the reinforcement particles prepared by the dual-coating process.

Keywords: C. Hardness; Metal matrix composites (MMCs); Microstructure; Mechanical property; Reinforcement particle

1. Introduction

Research into composites that incorporate ceramic particles as a reinforcement phase, namely metal matrix composites (MMCs), has aimed to enhance the wear resistance, hardness, and elasticity of pure metals [1,2]. Silicon carbide, alumina, titanium carbide (TiC), and titanium boride, all well-known ceramic materials with suitable mechanical properties, have been used as reinforcement phases for metal matrices. In particular, TiC has been widely used as a particulate-reinforcement phase for a matrix because of its high modulus, hardness, stiffness, and strength [3,4]. To maximize these advantageous effects of reinforcement particles in the matrix, the particles must be individually dispersed in the metal matrix without any aggregation. However, the reinforcement particles may self-aggregate both because of incompatibility between the TiC particles and the metal matrix and because of the relatively lower specific gravity of the TiC particle than that of the metal, thereby reducing the mechanical properties of the matrix despite the addition of the reinforcement particles. Therefore, for the homogeneous dispersion of TiC particles within the molten metal, the TiC particles have been coated onto an inoculant used in a iron (Fe)-based matrix to improve the crystal growth of graphite and to restrict the formation of cementite.

In this work, the reinforcement particles using the inoculant as a core particle were fabricated using a dual-coating process. In the first part of the process, TiC particles

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[*] Corresponding author. Tel.: +82 55 213 2742; fax: +82 55 262 6486.
[**] Corresponding author. Tel.: +82 55 213 3712; fax: +82 55 262 6486.
E-mail addresses: udam99@changwon.ac.kr (E.-H. Kim), jungyg@changwon.ac.kr (Y.-G. Jung).

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were coated with a nickel (Ni) phase to increase the compatibility between the reinforcement particles and the metal matrix. In the second part of the process, the Ni-treated TiC particles were coated onto the surface of the inoculant to improve the dispersibility of the reinforcement particles within the matrix. In particular, the Ni coating was conducted in an aqueous solution to increase the coating efficiency of Ni phase on the TiC particles. The efficiency could be controlled by the surface charge in the TiC particle, and increased by the strong attractive force between the Ni cations and the TiC particles in the condition with a large negative charge at a specific pH [5,6]. The microstructure and mechanical properties of the metal matrix composites with the addition amount of reinforcement particle were observed using various analytical techniques.

2. Experimental procedure

2.1. Materials and fabrication of the reinforcement phase

The inoculant, ferrosilicon (Fe–Si) of about 1–2 mm diameter provided by a commercial organization (Metia Co., Ltd., Changwon, Korea), was used as the core particle. TiC particles, with a nominal particle diameter of about 2 μm, were obtained from a commercial supplier (Korea Hydro & Nuclear Power Company, Seoul, Korea). $Ni(NO_3)_2$ was obtained from Sigma-Aldrich Korea (Yongin, Korea) to provide the Ni cation (Ni^{2+}) in an aqueous solution [7–9]. $Ni(NO_3)_2$ was a precursor for the Ni phase to be used as a coating agent in a concentration of 1 M.

In the first part of the process (Process I), TiC particles were sonicated in the aqueous solution of pH 12 for 24 h. $Ni(NO_3)_2$ with content of 1 M was then added to the solution with TiC particles and stirred at room temperature for 24 h. The prepared mixture was filtered and dried at 80 °C for a period of 24 h, and then heat-treated under H_2 atmosphere at 500 °C for 1 h. In the second part of the process (Process II), the prepared Ni-coated TiC particles and ferrosilicon powder were mixed with 1:10 and 3:10 in weight ratio, respectively, at room temperature, and an inorganic binder composed of tetraethyl orthosilicate (TEOS; Sigma-Aldrich Korea, Yongin, Korea) and sodium methoxide (NaOMe; Sigma-Aldrich Korea, Yongin, Korea) was then added. The mixture was dried at 80 °C for 24 h and heat-treated under H_2 atmosphere at 500 °C for 1 h. The prepared reinforcement particles were then injected into a molten Fe-based matrix at 1500 °C and cooled to room temperature. Conditions used to prepare the matrices with and without the reinforcement particles are given in Table 1.

2.2. Characterization

The morphology and microstructure of the synthesized reinforcement particles and the metal matrices with and without the reinforcement particles were observed using a scanning electron microscope (SEM; JEOL Model JSM-5610, Tokyo, Japan). The signals produced by the SEM were caused by secondary electrons. The crystal phase of the synthesized reinforcement particles was analyzed using an X-ray diffractometer (XRD; Philips X-pret MPD, Model PW3040, Eindhoven, Netherlands). The hardness values of the metal matrices with and without the reinforcement particles were measured using a Vickers indenter (HV-114, Mitutoyo Korea, Seoul, Korea). Tests were conducted using a load of 10 N at room temperature, and five or more measurements were made. The tensile strength, yield strength, and elongation of the metal matrices with and without the reinforcement particles were measured using a universal test machine (Instron Model 8500, Instron Corp., Canton, MA, USA). The abrasion resistance of the metal matrices with and without the reinforcement particles was carried out using an abrasion tester machine (R&B Model PD-102, Yasuda Seiki Seisakusho, Ltd., Nishinomiya, Japan) under the following conditions; rotation speed of 100 rpm, applied load of 5 kg, and testing time of 30 min.

3. Results and discussion

3.1. Preparation of the reinforcement particles

The Ni coating on the TiC particles is driven by the attractive force between the Ni cations and the TiC particles with negative charge, which depends on the pH of aqueous solution. The zeta potential of the TiC particle is increasingly negative with increasing pH value, implying that the adhesion between the Ni cations and the TiC particles is enhanced by the increase in the negative charge density of the TiC particle in the solution. However, in our previous work, it was already researched that $Ni(NO_3)_2$ powder is insoluble in the basic aqueous solution of pH above 12 [10]. This result indicates that the contact between the Ni cations and the TiC particles is obstructed resulting from the aggregation of $Ni(NO_3)_2$ sediments on the surface of TiC particle. Therefore, in this work, Process

Table 1
Conditions used to prepare reinforcement particles and mechanical properties of matrices with and without reinforcement particles.

Run number	Type of metal matrix composite	TiC/inoculant (wt%)		Hardness (GPa)	Tensile strength (MPa)	Yield strength (MPa)	Elongation (%)
Run 1	Conventional casting steel	N/A		1.47 ± 0.17	226 ± 11	190 ± 9.5	2.92 ± 0.146
	Inoculant-ceramic composite	TiC	Inoculant				
Run 2		1	10	1.91 ± 0.11	234 ± 12	184 ± 9.2	2.16 ± 0.108
Run 3		3	10	2.70 ± 0.06	250 ± 11	224 ± 7.8	0.75 ± 0.070

I was conducted in the aqueous solution of pH 12, as a marginal pH of soluble $Ni(NO_3)_2$ solution, to maximize the coating efficiency of Ni phase onto the TiC particles. The microstructure of the Ni-coated TiC particles is shown in Fig. 1(a), where the nano-sized spherical Ni particles (broken arrow) are homogeneously and individually coated on the surface of polygonal TiC particles (solid arrow) without any self-aggregation in spite of heat treatment. If the Ni particles are located on the TiC particles by simple shear force without the interaction between the TiC particles with negative charge and the Ni cations, the Ni particles could not be observed at the surface of TiC particle after heat treatment. In Process II, a commercial inorganic binder, composed of TEOS and NaOMe, was used as an adhesive reagent to increase the adhesion between the inoculant and the Ni-treated TiC particles. The NaOMe and TEOS are hydrolyzed and condensed during drying and heat treatment under air, being converted into sodium hydroxide and silica (SiO_2), respectively, in a white solid phase [11–13]. Note that the solidified inorganic binder firmly joins the inoculant and Ni-treated TiC particles. The Ni-coated TiC particles prepared are uniformly coated on the surface of the inoculant, as shown in Fig. 1(b), because of the solidification of the TEOS and NaOMe *via* the sol–gel reaction.

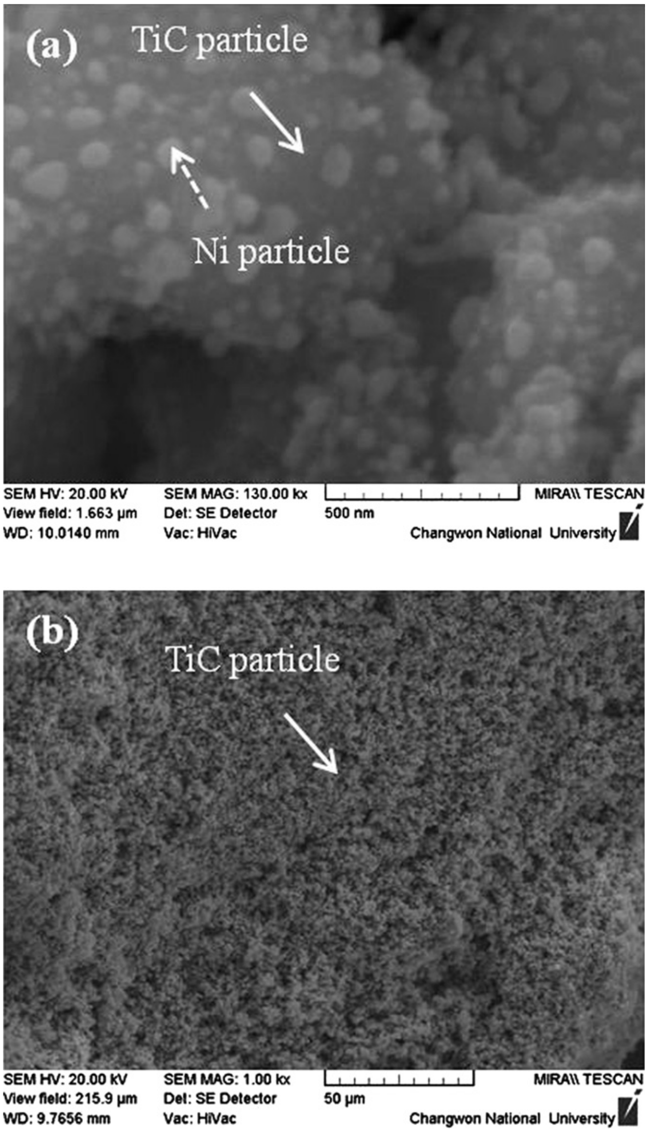

Fig. 1. SEM images of reinforcement particles prepared through (a) Process I and (b) Process II.

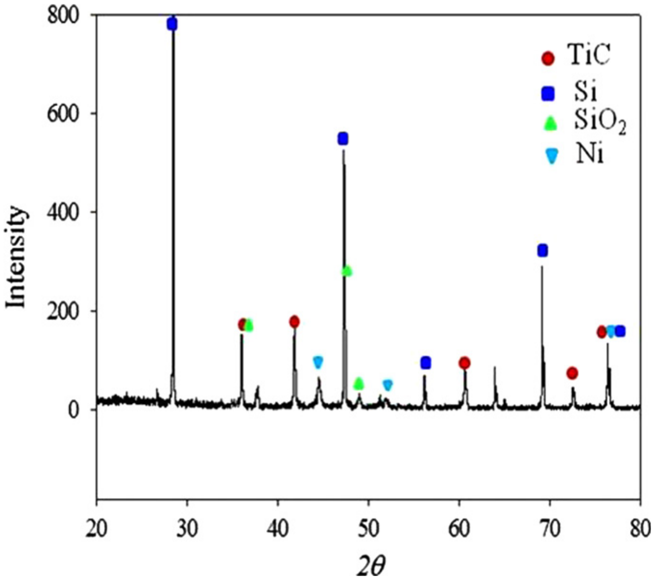

Fig. 2. XRD analysis of reinforcement particles prepared through the dual-coating process.

An XRD profile of the reinforcement particles prepared using the dual-coating process is shown in Fig. 2. The prepared particles consist of Si and SiO_2 phases from the inoculant and inorganic binder, together with the TiC and Ni phases, indicating that the reinforcement particles are well prepared by the dual-coating process. In addition, any deformation of the TiC and Ni phases after heat treatment, particularly oxidation, should be minimized to improve the reinforcing effect of the prepared particles. In this work, oxidation of the TiC and Ni phases was fortunately not observed. This indicates that heat treatment at 500 °C under H_2 is an effective way to prepare the reinforcement particles without transformation or degradation of the TiC and Ni phases.

3.2. Microstructure and mechanical properties of MMCs

The inoculants, used as core particles in this work, have often been added to a Fe-based matrix to accelerate the growth of graphite crystals and to restrain the generation of cementite. The microstructures of the prepared metal matrices are shown in Fig. 3, and the solid and broken arrows indicating ferrite and graphite, respectively. The shape and size of the graphite crystals generated in the Fe-based matrix are significantly influenced by the mutual competition between nucleation and growth. As the quantity of reinforcement particles dispersed in the matrix is increased, the graphite crystals become shorter and finer

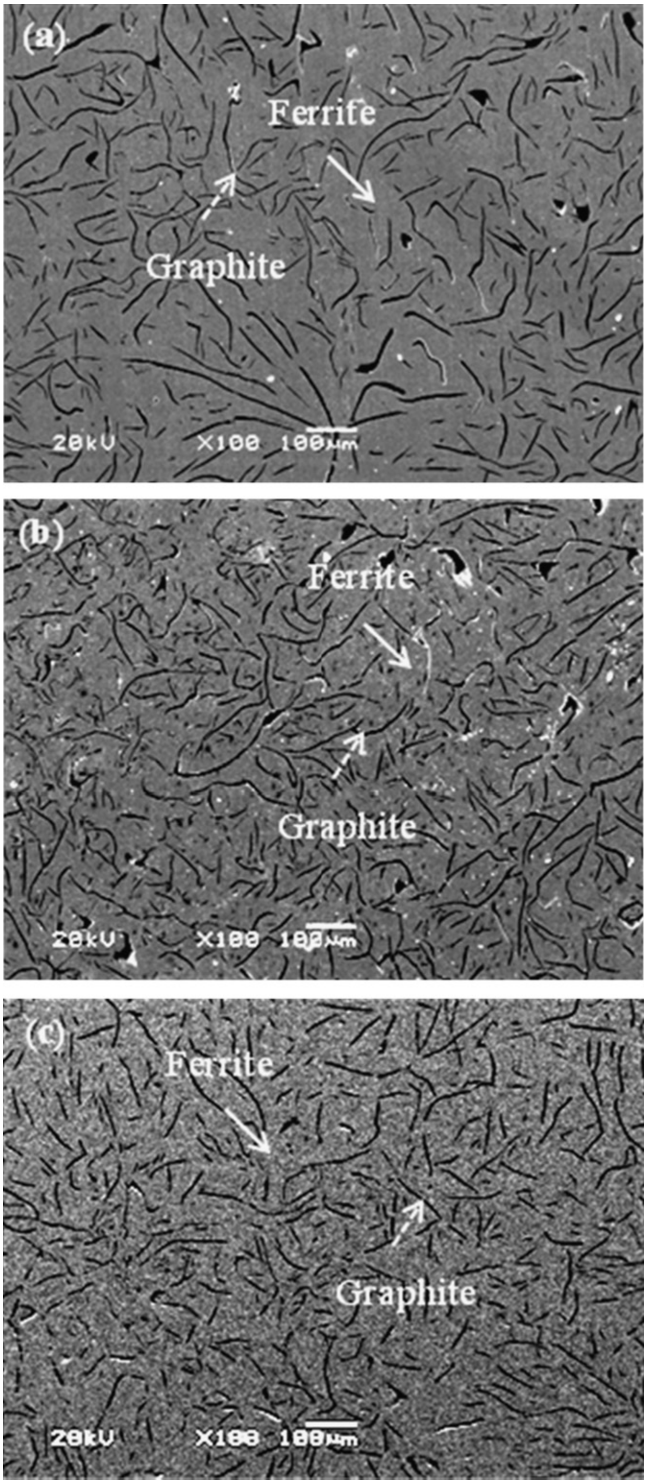

Fig. 3. SEM images of metal matrices containing (a) conventional casting steel (Run 1), (b) inoculant–ceramic composite with the 1 wt% TiC (Run 2), and (c) inoculant–ceramic composite with the 3 wt% TiC (Run 3).

(Fig. 3(b) and (c)). This is because the nucleation of graphite dominates its crystal growth, caused in turn by the increased number of TiC particles coated on the inoculant. However, in the conventional casting steel, local aggregation of the graphite phase is observable (Fig. 3(a)).

The various mechanical properties of the metal matrices prepared in this work are shown in Table 1. The hardness values of the reinforced matrices would ordinarily be higher than that of the conventional casting steel. The increase in the hardness value is induced by two reasons. First, the reinforcement particles with better mechanical properties prepared *via* the dual-coating process are homogeneously and uniformly dispersed in the metal matrix. Second, the reinforcement particles cause the graphite crystals to become small and fine, as shown in Fig. 3. When the number of graphite crystals is increased, the interface area between the graphite and the ferrite is increased. This interface inhibits dislocation movements from occurring due to plastic deformation of the matrix. That is, many small graphite crystals interfere with the propagation of deformation, leading to the increased hardness of the matrix with the reinforcement particles. The tensile and yield strengths of the 1 wt% TiC-reinforced matrix have similar values to those of the conventional casting steel; by contrast, for the 3 wt% TiC-reinforced matrix, the strengths are increased by the reinforcement effect of the TiC particles and the fine graphite, as shown in Fig. 3(C). In addition, the elongation of the matrix is conspicuously reduced by the addition of reinforcement particles, resulting from the increased strain resistance against stress by the TiC particles and the graphite crystals existed in the matrix.

The results for abrasion resistance are presented in Fig. 4, showing that the weight removed from the surface increases with the measuring time. It is found that the matrix with the 1 wt% TiC-reinforcement phase has higher abrasion resistance than the conventional casting steel, arising from the enhanced hardness caused by the reinforcing effect. However, for the matrix with the 3 wt% TiC-reinforcement phase, the abrasion loss is increased, compared with the conventional casting steel. It is because the 3 wt% TiC-reinforced matrix has the higher spin–slip property by the smaller and finer graphite generated on

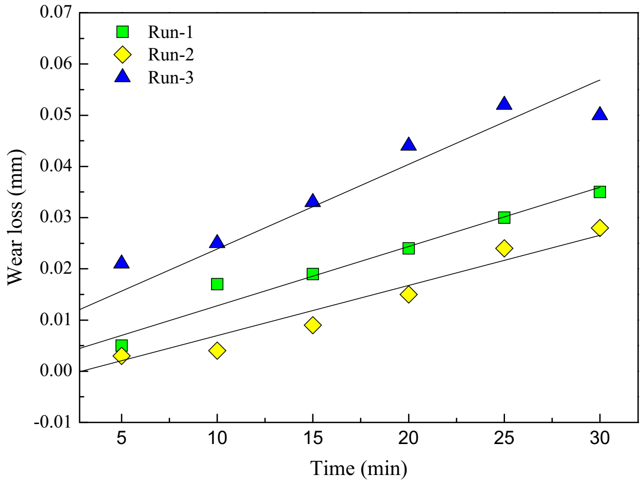

Fig. 4. Results of abrasion resistance measured for conventional casting steel and reinforced metal matrices.

the surface of matrix, compared with the matrices of Runs 1 and 2. Therefore, in the composite matrix prepared with the 3 wt% TiC-reinforcement phase, the abrasion resistance of matrix is lower than those without reinforcement particle and with the 1 wt% TiC-reinforcement phase because the heterogeneous boundary (the interface between the graphite and the ferrite) is easily impaired in the initial abrasive condition. In addition, a gradient of wear loss, particularly for the matrix with the 3 wt% TiC-reinforcement phase, increases significantly with measurement time. Therefore, a reasonable quantity of reinforcement particles in the metal matrix will provide desirable effects with respect to abrasion resistance.

Fig. 5 shows the coefficients of thermal diffusion and thermal conductivity for various metal matrices with and without the reinforcement phase. For a pure Fe matrix, they are 0.23 cm^2/s and 80.3 W m/°C at room temperature. The metal matrices prepared in this work show much lower thermal conductivity and thermal diffusion than those of the pure Fe matrix, because many graphite crystals distributed within the matrix hinder thermal diffusion.

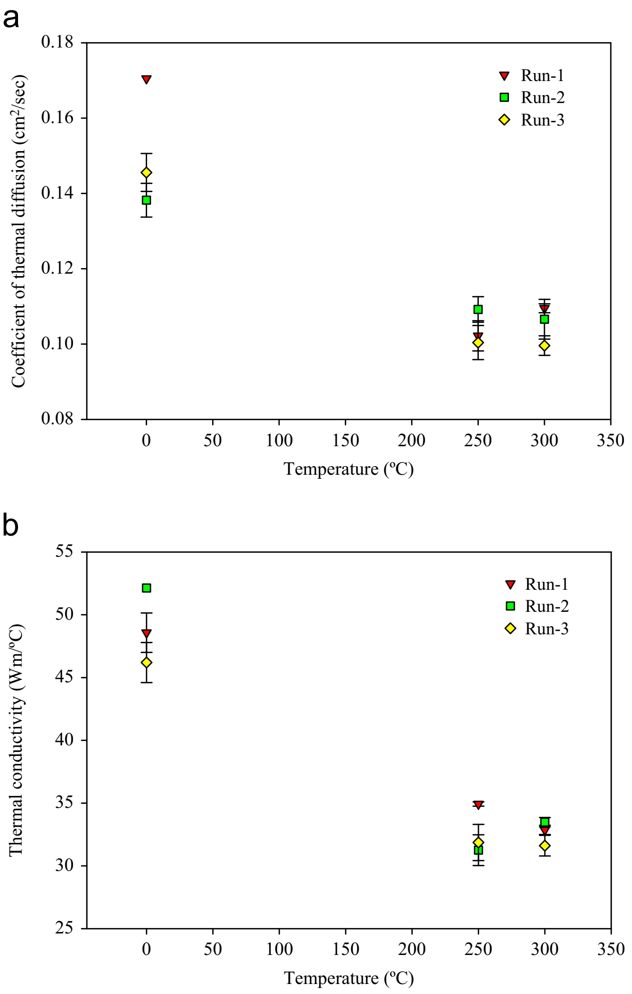

Fig. 5. Variation of (a) coefficients of thermal diffusion and (b) thermal conductivity for various metal matrices with and without reinforcement particles.

4. Conclusions

Reinforcement particles were successfully prepared through the dual-coating process, with the aim of improving the dispersibility and compatibility of the reinforcement particles in the metal matrix, leading to improvements in the mechanical properties of the matrix. In the first process (Process I), Ni particles are uniformly and homogeneously coated onto the surface of TiC particles in the aqueous solution of pH 12; this is achieved by the attractive force between the Ni cations and the TiC particles with negative charge. The Ni-coated TiC particles adhere well to the surface of the inoculant by the inorganic binder composed of TEOS and NaOMe solidified by the sol–gel reaction in the second process (Process II). The graphite crystals in the reinforced matrices are shorter and finer than that in the conventional casting steel, which implies that the nucleation of the graphite dominates its crystal growth because of the increased numbers of TiC particles coated on the inoculant. The 1 wt% TiC-reinforced matrix shows better hardness and abrasion resistance properties than the conventional casting steel. These results are related to the homogeneous dispersion of reinforcement particles with better mechanical properties within the matrix and the inhibition of dislocation propagation by the increased number of graphite crystals grown in the matrix. In the case of the 3 wt% TiC-reinforced matrix, even though the hardness and strength values are increased, the elongation and abrasion resistance properties deteriorate somewhat compared with the non-reinforced matrix; this is caused by the increased number of defect source such as the interface area between the graphite and the ferrite.

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