

plasma plume. To achieve a columnar porous structure as well as a high growth rate of the thicker pc-ZrO₂ layer, a pulsed 1 kW laser was applied. The irradiation parameters (pulse duration 1 ms, power density 10⁵ W/cm²) were chosen such that conventional thermal evaporation without plasma formation occurred. The targets for the ZrO₂ deposition were pressed and sintered from amperite powder (Y₂O₃: 7.79 wt%, HfO₂: 1.84 wt%, SiO₂: 0.20 wt%, ZrO₂: remaining).

Investigation of Boundary Layer Reactions: By means of PLD with a relatively high pulse energy of 800 mJ and stronger focusing, the generation of otherwise undesired droplets of the target material is forced. Since the directional characteristic of the droplets differs from that of the plasma plume, the substrate can be arranged in such a way that predominantly droplets are deposited. Due to the high kinetic energy of the droplets, they are flattened on impact before they solidify. The resulting Al and Ti hillocks are typically 2 to 20 μm in diameter and 0.5 to 5 μm high. (Fig. 2)

After droplet deposition, the Al/Cu and Ti/Cu samples were tempered (773 / 823 K, 100 / 300 min, atmosphere Ar 4.8, 10⁵ Pa). The resulting composition changes were examined by EDX in the SEM and also by AES depth analysis. The preparation of cross-sections of deposited droplets by ion milling with 7 keV Kr ions proved to be particularly useful (Fig. 3). At such cross sections, the chemical composition in the vicinity of the interface was measured by EDX.

Investigation of Roughness effects: Roughness values R_a of the copper substrate between 0.03 and 1.1 μm were prepared using appropriate grinding agents (abrasive paper, grit size P80 to P2500). The roughness was determined by a mechanical profiler (MAHR Perthometer S8P) and averaging over typically three samples polished in the same manner.

Bending bars with the dimensions 30 × 4.5 × 3 mm³ were coated onto one face with an area of 30 × 4.5 mm². These samples were then deformed by means of four-point bending where the coated surface was chosen as side face of the bending bar (cf. Fig. 4). Due to the bending deformation of the substrate, the coating is simultaneously subjected to a tensile load (Fig. 4, upper side) and a compressive load (lower side). To characterise the coating adherence quantitatively, the maximum substrate strains on the upper and lower sides of the bending bars were used. In particular, the critical spallation strains were determined where in the course of bending first spallings were visible to the naked eye under tensile and compressive load, respectively.

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Fabrication of Steel Matrix Composite Locally Reinforced with in situ TiB₂ Particulate using Self-Propagating High-Temperature Synthesis Reaction of Ni-Ti-B System During Casting**

By Hui-Yuan Wang, Qi-Chuan Jiang, Bao-Xia Ma, Yan Wang and Feng Zhao

Metal matrix composites (MMCs) are a class of materials that seek to combine the high strength and stiffness of a ceramic with the damage tolerance and toughness provided by a metal matrix.^[1,2] Although most of the studies on MMCs are centered on the lightweight engineering materials titanium, aluminum and magnesium due to their attractive combination of improved mechanical properties and weight-saving potential,^[3–5] there is also significant interest in developing particulate reinforced iron- and steel-based MMCs mainly to produce a combination of excellent wear resistance and/or cutting performance with improved toughness and to achieve significant cost reductions over existing materials, e.g. Co-WC

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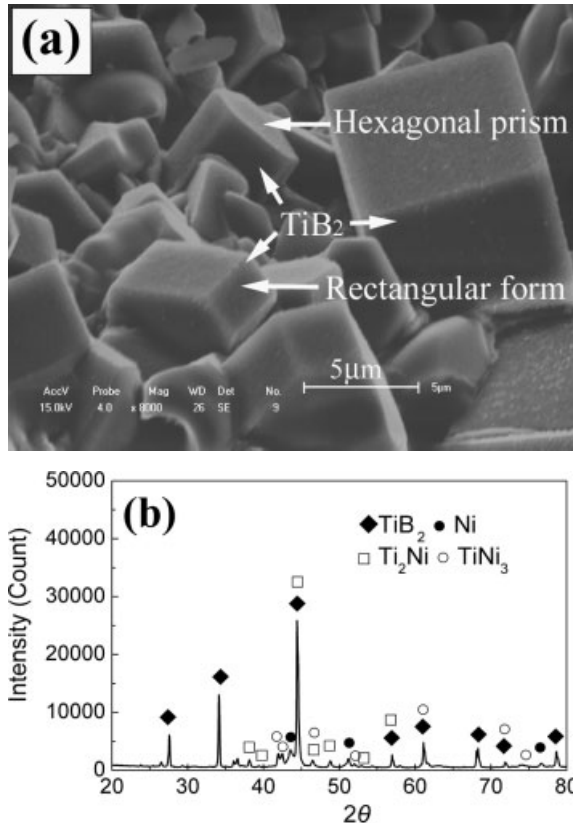


Fig. 1. a) SEM microstructure and b) XRD pattern of the SHS reaction products synthesized by 40 wt.% Ni–Ti–B system in a vacuum electrical resistance furnace.

cemented carbides.^[6–10] A majority of these composite materials focus on using TiC as the reinforcing particulate phase, primarily due to its high hardness, good wettability, low density, and its chemical stability with the iron and steel matrices.^[6–11] Recently, however, it has been found that the addition of TiB₂ to metal matrices can also greatly increase the stiffness, hardness and wear resistance. Furthermore, TiB₂ ceramic possesses many desirable properties, such as high melting temperature (2980 °C), high hardness (33 GPa), high modulus (530 GPa) and low density (4.45 gcm⁻³), as well as a significantly lower coefficient of thermal expansion than steel ($\sim 7.2 \times 10^{-6} \text{ K}^{-1}$ for TiB₂ and $\sim 13.0 \times 10^{-6} \text{ K}^{-1}$ for steel).^[9,12,13] These outstanding features portray it to be a good potential reinforcing candidate in metal matrix. In addition, it is considered that TiB₂ is the best reinforcement for achieving high specific strength and high specific modulus in the iron and steel matrices.^[9]

Generally, the TiC- and TiB₂-Fe composites have been produced by various processing routes such as powder metallurgy (P/M),^[2,14] combustion synthesis, also called self-propagating high-temperature synthesis (SHS),^[11,15] conventional melting and casting technologies, i.e. in situ reactions between Ti powder and C-rich liquid Fe,^[16] C powder and liquid Ti–Fe alloys^[17] or C–Fe and Ti–Fe liquid alloys,^[18] direct dispersion of TiC powder,^[19,20] TiC–Fe^[21] or TiB₂–Fe^[9,10] master alloys in liquid steel, and carbothermic reduction pro-

cesses.^[6,7,22] The P/M is usually energy and capital intensive and prove difficult to scale up for large components. As the reinforcing phase was prepared separately prior to the composite fabrication, the scale of the reinforcement is limited by the starting powder size, which is typically of the order of microns to tens of microns. Other main drawbacks that have to be overcome are the interfacial reactions between the reinforcements and the matrix, and poor wettability between the reinforcements and the matrix due to surface contamination of the reinforcements. Currently, it is well established that SHS has many attractive advantages such as high purity of product, low processing cost, and efficient in energy and time.^[3] However, one of the initial drawbacks of the SHS reactions for industrial applications has been that the final product can be highly porous, e.g. typically 50% of theoretical density.^[23] Therefore, this process must be combined with a densification step, such as hot-pressing, extrusion, quasi-isostatic pressing (QIP), modified HIP-ing or shock-wave compaction.^[24] Unfortunately, such techniques are too expensive or complex to be accepted by the engineering community for general applications. It is well known that casting process is the most effective way to obtain low cost MMCs in conventional process. However, it is difficult to fabricate the MMCs with a high volume fraction of reinforcement, e.g. with a reinforcement > 10 vol.%, due to the poor castability.

Moreover, most of the TiC- and TiB₂-Fe MMCs in the previous studies are monolithic composites, while less work has been carried out on the locally reinforced Fe MMCs.^[25–27] However, from the point of view of the application, the local wear-resistance of components often determines their useful life, and, it is desirable that the local region of casting rather than all the casting is reinforced by the relatively costly particulates.^[27] Furthermore, the locally reinforced composite also offers a virtually unique combination of various properties, for example, the strength, toughness, hardness and wear-resistance etc.

In the present study, the feasibility of fabrication of steel MMCs locally reinforced with TiB₂ particulates synthesized by the SHS reaction of Ni–Ti–B system which is ignited by the heat of molten steel during casting was investigated. The purpose of the use of the SHS and traditional casting routes provides a promising process for fabrication of steel MMCs because of its significant inherent simplicity and near-net shape processing capability. It is expected that the preliminary results can be significant in promoting the development of the locally reinforced steel matrix composites.

In order to study the products of SHS reaction in Ni–Ti–B system, the preforms were placed in a vacuum electrical resistance furnace to heat to about 600 °C, and then the bottom surface of the preform was ignited by applying a current of about 15 A through a resistance wire. The SEM microstructure and XRD pattern of the SHS reaction products synthesized by a 40 wt.% Ni–Ti–B system are shown in Figures 1a and 1b, respectively. XRD result reveals that, in addition to the Ni and TiB₂ phases, a small amount of Ti₂Ni and TiNi₃ phases are also

detected in the reaction products, which indicates that the full conversion of Ti to TiB₂ has not been achieved. In this case, the possible SHS reactions may be summarized as an overall reaction:



It is worth noting that the transient TiNi_x phases should not be present in the end products, since the Ni and TiB₂ are the thermodynamically stable phases in the SHS reaction products. Gotman et al.^[28] have synthesized Al MMCs reinforced with 30 vol.% TiB₂, TiC and TiC+TiB₂ ceramic reinforcements via the SHS reaction of Al-Ti-B (C, B₄C or C+B) systems, respectively, followed by high-pressure consolidation to full density. In their study, XRD result reveals that in all the systems except Al-Ti-C, strong additional peaks of the transient TiAl₃ are also observed. They explained that, under the conditions of a propagating combustion wave, the dwell time of the material in the combustion zone is not long enough to achieve the equilibrium compositions.^[28] This proposal can also be extended to the Ni-Ti-B system in the present study to explain the formation of transient TiNi_x. In fact, the reaction kinetics determines which phase will form during the SHS reaction.^[5,29] It has been reported that the transient TiAl₃ phase can be effectively suppressed by the incorporation of an appropriate amount of C or an excess of B into the Al-Ti (TiO₂)-B systems.^[30-33] Therefore, the research on eliminating transient TiNi_x in Ni-Ti-B system by the addition of C or excess B is being carried out in our group. The results will be described in detail elsewhere. It can be seen from the SEM micrograph (Fig. 1(a)) that the TiB₂ is present typically as a hexagonal prism or a rectangular form in the products of Ni-Ti-B system. It is interesting to note that these morphologies are quite similar to those observed in the SHS reaction products of Al-Ti-B system.^[32] It is believed that the TiB₂ morphologies must be from its close-packed hexagonal crystal structure.

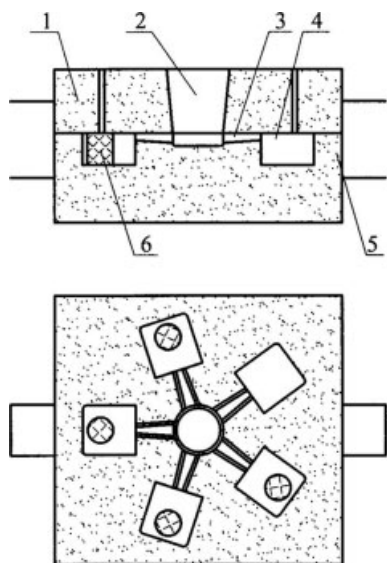


Fig. 2. Schematic diagram of green preforms in the sand mold (1. Upper sand mold; 2. Sprue; 3. Flow gate; 4. Casting; 5. Lower sand mold; 6. Preform.)

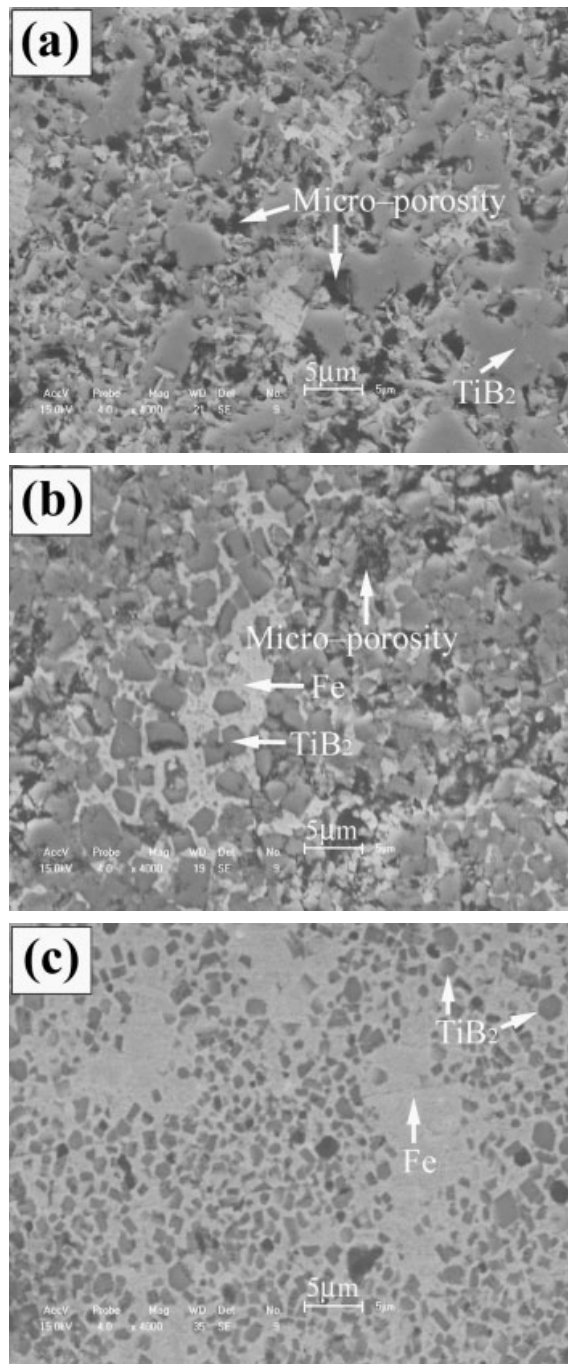


Fig. 3 Typical SEM microstructures of the medium steel matrix composites locally reinforced with in situ TiB₂ particulates synthesized by SHS reaction of Ni-Ti-B systems with a) 20, b) 30 and c) 40 wt. % Ni, respectively.

When the molten steel was poured into the sand mold, as schematically illustrated in Figure 2, the SHS reaction in the Ni-Ti-B system was ignited by the release heat of the steel melts. Subsequently, the molten steel will spontaneously infiltrate into the pores, resulting from the gas evaporation that is generated by high exotherm of the SHS reaction, in the reacting preform driven by the capillary force. After solidification, the pores infiltrated with steel melts in the reacted preform will evolve into the Fe-rich region.

Figures 3 and 4 show the SEM microstructures and XRD patterns of the locally reinforced medium steel matrix composites fabricated by using the SHS reaction of Ni–Ti–B systems with various Ni content, respectively. XRD results reveal that the composites consist of Fe, Ni and TiB₂, as well as a small amount of TiC and Fe₂B phases. However, no TiNi_x compound is found in the XRD result. The reason may be that its low content and intensity due to the dilution by the matrix material could not be detected by XRD analysis. According to,^[9,10] the TiC and Fe₂B phases may be formed by the following reactions:



It should be mentioned that the [C] results from the presence of 0.45 wt.% C in the medium carbon steel matrix. The formation of [Ti] and [B] will be discussed later. However, the overall Reaction 3 would not proceed without the [C] participation, since it is thermodynamically unfavorable for a direct reaction between TiB₂ and Fe to form Fe₂B ($\Delta G \gg 0$).^[9] Although the reaction (3) is very weakened due to the relatively low intensities of TiC and Fe₂B phases detected by XRD (Fig. 4), it can also be concluded that the TiC is thermodynamically stable than TiB₂ in Fe–C alloys.

Microstructural characterization shows the presence of many macro-pores and blowholes in the locally reinforced region only in the composite synthesized by the 10 wt.% Ni–Ti–B system. Although the presence of significant micro-porosity can be detected in the composite synthesized by 20 wt.% or 30 wt.% Ni–Ti–B system, the amount of it decreases with the increasing of Ni content, as shown in Figures 3a and b, respectively. Furthermore, it is interesting to note that only the presence of minimal micro-porosity can be found in the composite synthesized by 40 wt.% Ni–Ti–B system, which reveals that the near-fully dense composite locally reinforced with high volume fraction of the in situ TiB₂ particulate can be fabricated, as shown in Figure 3c. As the Ni content increases in

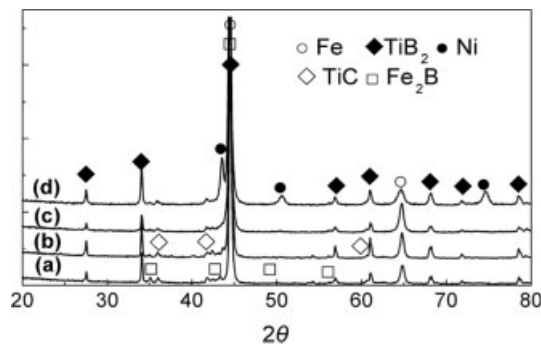


Fig. 4. XRD patterns of the medium steel matrix composites locally reinforced with in situ TiB₂ particulates synthesized by SHS reaction of Ni–Ti–B systems with a) 10, b) 20, c) 30 and d) 40 wt.% Ni, respectively.

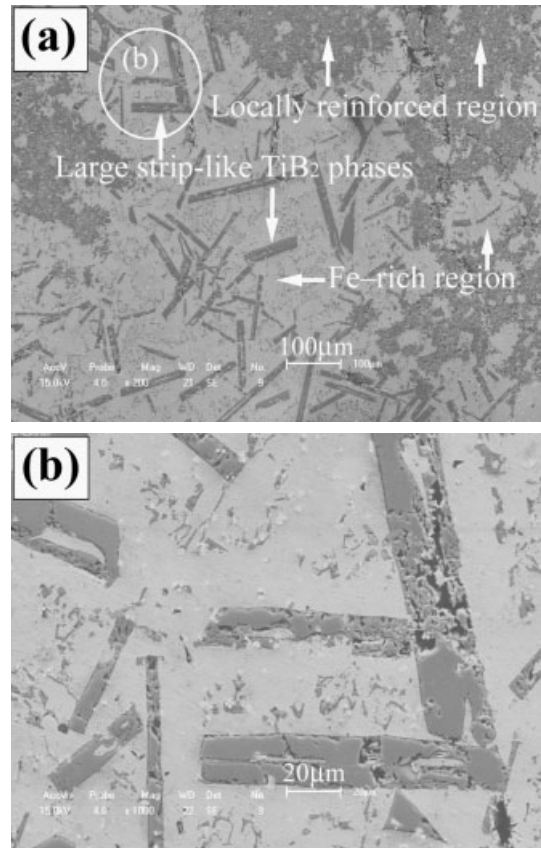


Fig. 5. a) The in situ formed TiB₂ with a strip-like morphology in the locally reinforced medium steel matrix composites fabricated by the SHS reaction of 20 wt.% Ni–Ti–B systems and b) high magnification of a).

Ni–Ti–B system, in addition to participating in the reactions, Ni also serves as a diluent, and therefore, the reaction between Ti and B is relatively weakened by the presence of an excess of Ni. Furthermore, the Ti and B reactants per unit volume in the system are also reduced, which results in the decreasing of the heat generation during the SHS reaction. As a result, the gas evaporation generated by the SHS reaction is reduced, resulting in the decreasing of the macro- and micro-porosity in the locally reinforced region in composites.

It can be observed from Figures 3a–c that the distribution of TiB₂ in the locally reinforced region in composite synthesized by 20 wt.% or 30 wt.% Ni–Ti–B system exhibits a certain amount of conglomeration; however, that in the locally reinforced region in composite synthesized by 40 wt.% Ni–Ti–B system reveals a relatively homogenous distribution. It can also be seen that the size of TiB₂ particulate decreases with the increasing of Ni content in the Ni–Ti–B system. This may be attributed to the decrease of the combustion temperature because crystal growth is an exponential function of the combustion temperature.^[34,35] Furthermore, the diluting effect of Ni also increases with the increasing of Ni content, which may hamper the grown-up of TiB₂ crystals during the SHS reaction.

Comparing Figure 3c with Figure 1a, we can find that, although, the Ni content (40 wt.%) is the same in the Ni–Ti–B

Table 1. Hardness and wear resistance values of the medium steel, high Cr hot work steel and composites locally reinforced with in situ TiB₂ particulates.

Materials	Medium steel matrix	High Cr hot work steel	Locally reinforced composites (Ni content in Ni-Ti-B system, wt.%)		
			(20)	(30)	(40)
Hardness (HRC)	<20	52.0	30.5	38.2	44.5
Wear rate ($\times 10^{-10}$ m ³ /m)	4.5245	2.582	-	1.7562	1.4235

system, the TiB₂ particulate size in the steel matrix composite fabricated by the SHS reaction during casting (~ 3 μm) is significantly smaller than that in the products synthesized by the SHS reaction in a vacuum electrical resistance furnace (~ 8 μm). It can be explained that much of heat released by the SHS reaction dissipates rapidly into the steel melt owing to the direct contact between the reacting preform and the steel after the reaction was ignited in the green preform by the heat of the molten steel. However, the propagation of heat in the vacuum surroundings is relatively slower, and therefore, the holding time of the reacting or reacted preform in high temperature is relatively long, resulting in the TiB₂ crystal growing into large particulate.

Figures 5a and b show the SEM micrographs of the in situ formed TiB₂ with a strip-like morphology and its high magnification in the locally reinforced medium steel matrix composites fabricated by the SHS reaction of 20 wt.% Ni-Ti-B systems, respectively. Generally, the large strip-like TiB₂ is distributed in the Fe-rich region located in the locally reinforced region or in the steel matrix region nearby the interface between matrix and reinforced region. It is believed that the TiB₂ particulate in the locally reinforced region is synthesized mainly by the SHS reaction of the Ni-Ti-B system, and its detailed morphology can be seen in Figure 3a. Therefore, this process can be summarized as an overall Reaction 1. Due to suffering from the high temperature in the combustion zone, some Ti and B particles will dissolve in steel melts and form the [Ti] and [B]. During solidification, the Ti and B atoms will form the TiB₂ nucleus, which further grows into the large crystal with the strip-like morphology due to the relatively high solute concentration and long enough solidification time of the steel melts. Therefore, the reactions can be summarized as follows:



The hardness and wear resistance values of the medium steel matrix, high Cr hot work steel (10.41Cr-0.33C-1.38Si-

1.13Mo-Fe balance, all in wt.%) that is famous for its high hardness and high wear resistance, and composites locally reinforced with in situ TiB₂ particulates are shown in Table 1. It can be seen that, although, the hardness values of the composites are significantly higher than that of medium steel and lower than that of high Cr steel, the volumetric wear losses of the composites are lower than those of the medium steel and high Cr steel. Generally, the abrasive wear resistance can be defined as the inverse of volume loss. As a result, it is interesting to conclude that the wear resistances of the composites are higher than those of the medium steel matrix and high Cr steel even though it has a significantly higher hardness. Improvement of wear resistance of the locally reinforced composite is mainly attributed to the presence of high volume fraction of hard TiB₂ ceramics. With the increasing of Ni content in Ni-Ti-B system, although, the size and volume fraction of the TiB₂ particulate in the locally reinforced region of the composite are decreased, the hardness and wear resistance values of the composites are increased, as shown in Table 1. This is due to the reducing of micro-porosity in the composite.

The medium steel matrix composites locally reinforced with in situ TiB₂ particulates synthesized by the SHS reaction of Ni-Ti-B system during casting are successfully fabricated. Microstructural characterization shows that the size of TiB₂ particulate and amount of micro-porosity in the composite are decreased with the increasing of Ni content in the Ni-Ti-B system. Although most of fine TiB₂ particulates which form by the SHS reaction of the Ni-Ti-B system are present in the locally reinforced region, some large strip-like TiB₂ phases which form during solidification process are distributed in the Fe-rich region located in the locally reinforced region or in the steel matrix region nearby the interface between matrix and reinforced region. The hardness values of the composites are significantly higher than that of medium steel and lower than that of high Cr steel; however, the wear resistances of composites are higher than those of medium steel and high Cr steel even though it has a significantly higher hardness. Furthermore, the hardness and wear resistance values of the composites increase with the increasing of Ni content in the Ni-Ti-B system due to the reducing of micro-porosity in the composite.

Experimental

The starting materials in the present study were made of commercial powders of 10, 20, 30 or 40 wt.% Ni (99.8 % purity, ~ 5 μm), Ti (99.5 % purity, ~ 15 μm) and B (98.0 % purity, ~ 3 μm). Titanium and boron powders were at a ratio corresponding to that of stoichiometric TiB₂. Powder blends were mixed by a ball milling for 8 h, and then were uniaxially pressed into cylindrical preforms (20 mm diameter and 15 mm length) under pressures ranging from 75–80 MPa to obtain densities of 68 ± 5 % theoretical density.

Medium carbon steel (0.45C–0.42Si–0.30Mn–Fe balance, all in wt.%) was selected as the matrix for the composite, and the steel melt was prepared in a 5 kg medium-frequency induction furnace in air environment. After being dried in a vacuum oven at about 300 °C for 3 h to remove any trace of moisture, the green preforms were placed and fixed on the bottom of the sand mold, as schematically illustrated in Figure 2. Subsequently, the molten steel with a temperature of about 1600 °C was poured into the mold to ignite the SHS reaction in the preforms. After solidification and cooling, the matrix steel and composite castings were removed and sectioned in the side position.

The sliding abrasive wear rates of the materials were tested under loads of 35 N using a pin-on-disc apparatus. All the materials were used as pin samples with 6 mm diameter and 12 mm height, and SiC abrasive papers of 600 grit size were used as the counterface. Metallographic samples were prepared in accordance with standard procedures used for metallographic preparation of metal samples, and etched with 5 vol.% HNO₃ solution for 5–10 s at room temperature. Microstructure and phase analyses of the samples were investigated by using scanning electron microscopy (SEM) (Model SHIMADZA SSX-500, Japan) equipped with energy-dispersive spectrum (EDS) (Model SHIMADZA, SEDX-500, Japan) and X-ray diffraction (XRD) (Model D/Max 2500PC Rigaku, Japan)

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Cyclic Deformation Characteristics of Titanium-Matrix Composite Reinforced with In-situ TiB Whiskers**

By Sie Chin Tjong* and Guisong Wang

Titanium and its alloys are important and useful class of engineering materials because of their excellent combination of strength and fracture toughness as well as low density. However, these materials are not suitable for application at high temperatures due to their high oxidation rates and loss of strength.^[1] There is considerable interest in the development of Ti-based metal-matrix composites (TMCs) reinforced with high-stiffness ceramic reinforcements, especially for application in the aerospace and automobile industries. The incorporation of ceramic reinforcements in Ti results in a considerable improvement in its mechanical properties and service temperature. The reinforcements could be in the form of fibers, whiskers or particles. Continuous SiC fibers have been used to reinforce Ti alloys in the past few years.^[2,3] However, chemical reactions occur between the fiber and metal matrix during the processing, leading to the formation of the reaction products such as Ti₃SiC₂, TiC_x and TiSi₃(C) products at the fiber-matrix interface. This leads to a degradation of the mechanical strength of TMCs.^[4,5] Moreover, mechanical and physical anisotropic properties are another issues encountered using continuous SiC fiber reinforcement. An effective way to

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