Synthesis and characterization of austenitic Steel matrix composite reinforced with in-situ TiC particles

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Hard particles like carbides, oxides, nitrides, and carbonitrides reinforced steel matrix composites are known for their excellent abrasive wear resistance [1]. Powder metallurgy techniques are often used for the synthesis of these composites [1–3]. However, materials produced through this route generally suffer from the problems of porosity and contaminated matrix-reinforcement interfaces. Therefore, liquid-based techniques involving the *in-situ* generation of the reinforcing phase have emerged as a preferred synthesis route for these materials [4–12]. Das *et al.* has given a comprehensive review on the various synthesis routes of TiC-reinforced iron-based composites [13]. The objective of this paper is to study the feasibility of processing *in-situ* TiC reinforced austenitic matrix composite utilizing siliceous sand (~73% Fe_2O_3, ~9% TiO_2, ~6% Al_2O_3, ~4% SiO_2, ~3% Na_2O and balance Cr_2O_3, V_2O_5 and NiO impurities (all in wt.%)) as the main raw material and Al as the reducing agent. Microstructural stability of the composite as well as its wear behavior have been investigated.

The charge calculation for the synthesis of composite was done by simple stoichiometric method aiming 13 vol.% TiC in Fe-0.8% C-14% Mn matrix. Both cast iron (3.86% C, 1.25% Si, 0.2% Mn, 0.3% P and balance Fe (in wt.%)) and graphite were used as the source of C. Anticipating the loss of C due to oxidation during SHS reaction, 50 wt.% excess C was added in the form of graphite. Although TiO_2 is present in the siliceous sand, some laboratory grade fine TiO_2 powder was also added in the charge material to get the desired volume fraction of TiC. Stoichiometric amount of Al, needed to reduce all the oxides, was added in the charge. The sand was washed with hot water to discard the deleterious Na_2O, which may cause problem in slag-metal separation. The charge mixture consisting of 440 g sand, 20 g TiO_2, 200 g cast iron, 10 g graphite, 130 g Al and 60 g Fe-Mn was taken in a zircon painted clay graphite crucible and heated to 1023 K in a resistance furnace. After about 15 minutes of soaking, the crucible was taken out and fine Mg turnings were added to trigger the reduction reactions. The liquid metal was subsequently solidified in a metallic die by a bottom pouring arrangement. The compositions of as-cast composite and Hadfield Mn-steel are given in Table I. Metallographic samples of dimension 12 × 12 × 10 mm were cut from the middle portion of the castings. The specimens were polished according to the standard metallographic technique and etched with 2% Nital. They were examined in the secondary electron imaging mode at 20 kV in a scanning electron microscope (JEOL, JSM-5800) equipped with an energy dispersive X-ray spectrometer (EDS). The various phases revealed by metallography were analyzed using CoK_α or FeK_α radiation in a Philips X-ray diffractometer (PW-1710) equipped with a solid-state semiconductor detector and the scanning speed was 1.2°/min. The tube voltage/current for Co and Fe targets were 40 kV/20 mA and 30 kV/10 mA, respectively. Abrasive wear tests were carried out on 12 mm × 12 mm cross-section samples having a thickness of 10 mm, against a 220 grit SiC paper affixed to a rotating flat disc of 250 mm diameter [14]. The sliding velocity was fixed at 2.61 ms^{-1} and track diameter was 100 mm. All the experiments were carried out at a load of 9.8 N. Each experiment was repeated three times. Wear rates of the specimens were computed by the weight loss technique.

The SEM micrographs of the as-cast composite, Fig. 1a and b, reveal the presence of TiC particles in almost a single-phase matrix. Various phases observed in the microstructure have been labeled in Fig. 1b. XRD plot of composite (Fig. 2) indicates the presence of strong peaks of austenite with very weak peaks of ferrite. The volume fractions of austenite, ferrite and TiC, as measured by image analysis, are 80, 10;, and 9–10%, respectively.

Fe_2O_3, TiO_2, and SiO_2 present in the sand react with Al and get reduced to Fe, Ti and Si, respectively. Iron, thus generated, forms the matrix along with C, Al, Si and Mn. EDX analysis shows that matrix of the composite contains a substantial amount of Al (~6%). This is due to the fact that some part of Fe_2O_3, TiO_2, and SiO_2 of the charge is lost in the slag, i.e., they do not take part in the reduction reaction. Hence, part of Al remains in solution and joins the matrix phase. Matrix also contains Mn as ferro-manganese, which is added to stabilize austenite. Reduced titanium reacts with C to form *in-situ* TiC particles.

In order to assess the stability of austenite matrix, as-cast composite was annealed at 900 C for different

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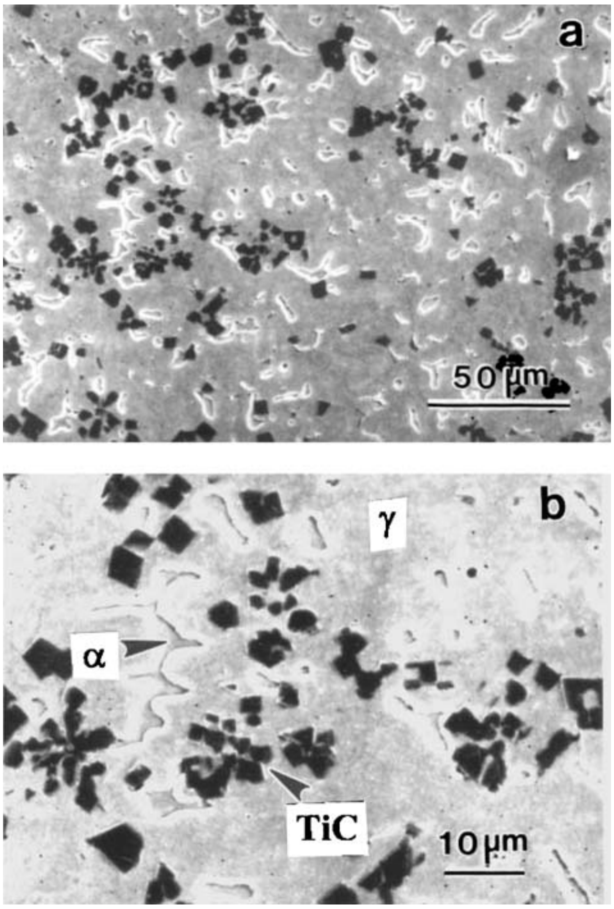

Figures 1 (a) and (b) SEM micrographs of as-cast composite.

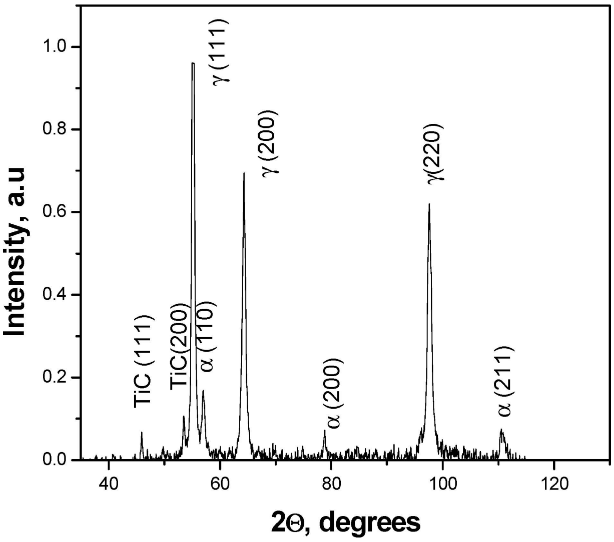

Figure 2 XRD of as-cast composite using Fe-Kα radiation.

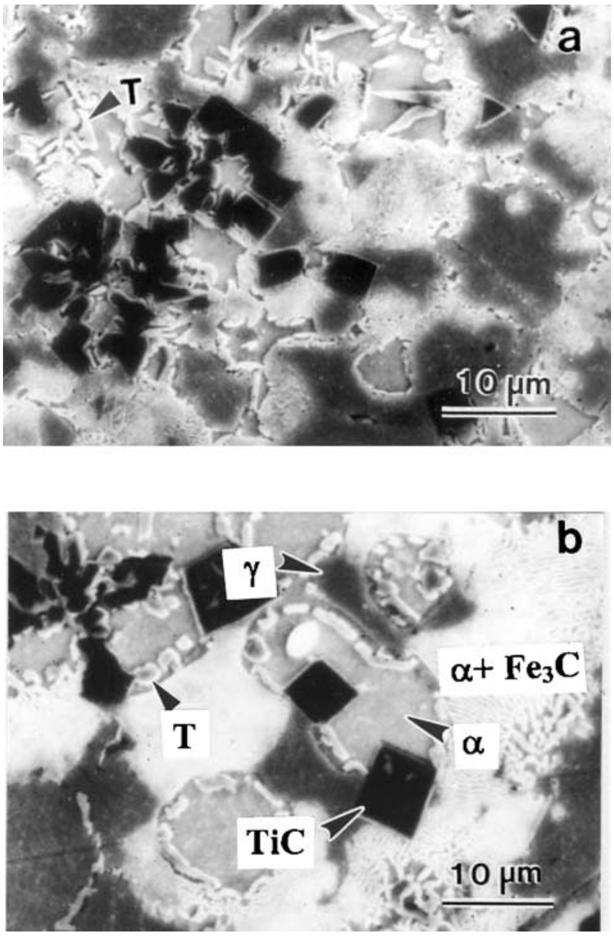

Figure 3 SEM micrographs of composite, annealed at 900 C for (a) 3 hr and (b) 6 hr.

times, namely 1, 3 and 6 hr. It is quite evident from the SEM micrographs of the annealed composites (Fig. 3a and b) that on annealing the austenite-matrix of the as-cast composite decomposes. The TiC particles remain almost unaffected. The changes in X-ray diffraction pattern with time during annealing at 900 C (Fig. 4) reveal that the volume fraction of austenite decreases and that of ferrite increases. The different phases, as observed in the annealed microstructure, have been labeled in Fig. 3b. A new phase, marked as T in the micrograph, appears after 3 hr of annealing and has been identified as Fe_2AlMn on the basis of composition. For comparison, Hadfield Mn-steel was also annealed at 900°C for 3 and 6 hr. The X-ray diffraction patterns of the as-cast and annealed (900°C for 6 hr) Hadfield-Mn steel (Fig. 5) indicates that no phase-decomposition occurs in Hadfield Mn-steel. The decomposition of austenite, in the case of composite, may be due to the fact that the matrix of the composite contains a substantial amount of Al (∼6%). During annealing Fe_2AlMn forms, resulting in a decrease in Mn content of the matrix, and thereby transforming austenite to ferrite. However, it appears that formation of Fe_2AlMn is not complete after 6 hr of annealing. As a result, some of the austenite still remains in the microstructure. It is quite likely that on further cooling some of the austenite, which is lean in Mn, decomposes to ferrite and cementite. On the other hand, Hadfield Mn-steel contains only 0.12% Al. Another factor leading to the formation of ferrite could be the non-stoichiometric nature of TiC particles in the composite. The microhardness and lattice parameter of TiC in the as-cast composite are 1800–2200 HV and

TABLE I. Chemical compositions of as cast composite and Hadfield Mn-steel

Material	wt.%Al	wt.%Mn	wt.%Si	wt.%Ti	wt.%C	wt.%Fe
Composite	5.12	13.56	1.93	5.65	2.4	Balance
Hadfield Mn-steel	0.12	13.86	0.25	–	1.42	Balance

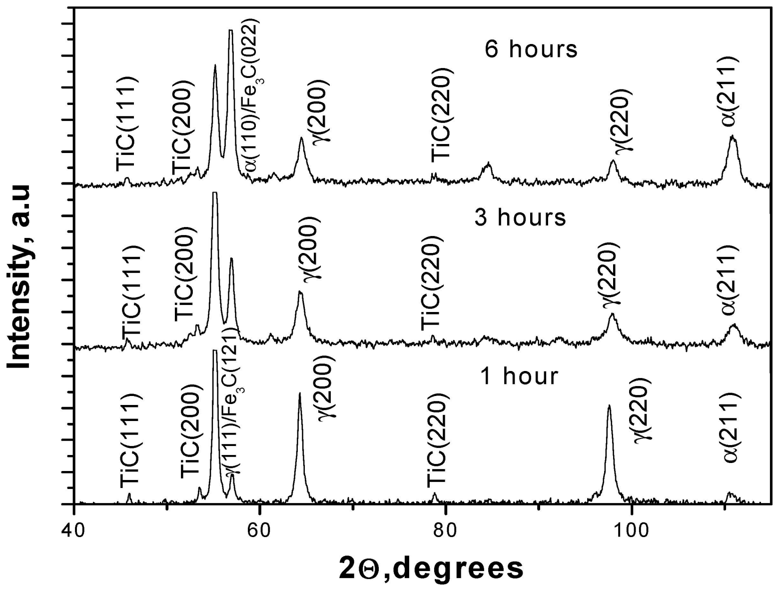

Figure 4 XRD patterns of composite annealed for different times using Fe-Kα.radiation.

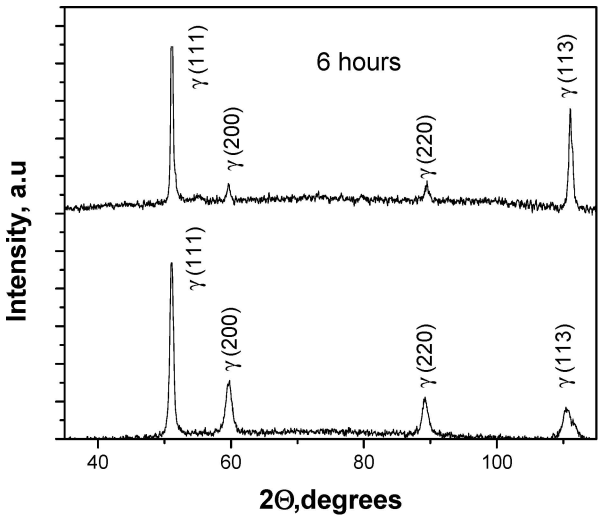

Figure 5 XRD patterns of Hadfield Mn-steel in as-received and annealed (900 C, 6 hr) condition using Co-Kα radiation.

0.4320 nm, respectively [15]. The reported microhardness and lattice parameter of stoichiometric TiC are 2900–3200 HV [16] and 0.4327 nm [17]. The microhardness and lattice parameter measurements indicate that TiC particles are non-stoichiometric in nature. It has been found by the present authors [18] that TiC particles in the Fe-TiC composites with ferrite, ferrite-pearlite, pearlite and pearlite-cementite matrix, synthesized by aluminothermic reduction, decrease the degree of non-stoichiometry either by taking C from the matrix or by rejecting Ti from TiC, which depends on the C content of the matrix. In the present study, it appears that C diffuses from matrix to TiC to decrease the extent of non-stoichiometry of TiC resulting a decrease in C content of the matrix, and thereby transforming austenite to ferrite. The presence of higher amount of Al and Si in the matrix of as-cast composite compared to the same in Hadfield Mn-steel is also a contributing factor in the formation of ferrite, since Al and Si are both ferrite stabilizers.

The hardness of the as-cast composite is higher than the Hadfield Mn-steel (Table II). This is due to the presence of TiC in the composite. Fig. 6 shows that the hardness of the composite increases, whereas the hardness of the reference material remains almost constant with annealing time. The increase in hardness of the composite is due to the precipitation of Fe_2AlMn phase followed by an increase in volume fraction of Fe_2AlMn phase. Decomposition of austenite to ferrite and cementite also contributes to the increase in hardness. The hardness of the Hadfield Mn-steel remains constant, as there is no change in the microstructure.

The abrasive wear resistance property of as-cast composite has been compared with a Hadfield Mn-steel (Fig. 7). It is evident from Fig. 7 that material loss is more in case of Hadfield Mn-steel compared to as-cast composite. This is due to the fact that TiC particles act as main load bearing constituents and re-

TABLE II. Hardness of as-cast composite and reference material

Material	Hardness (R_C)
Composite	36
Hadfield Mn-steel	24

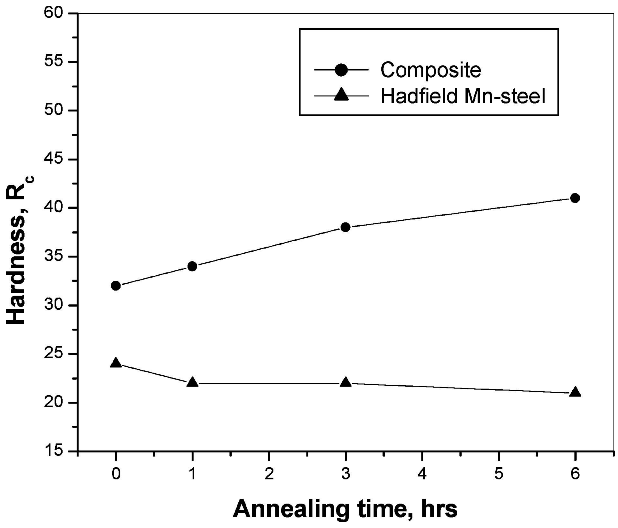

Figure 6 Change in hardness with annealing time at 900 C.

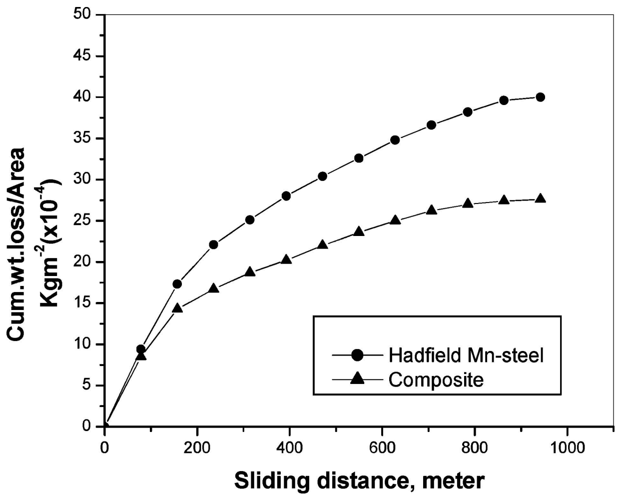

Figure 7 Wear behaviour of as-cast composite and Hadfield Mn-steel.

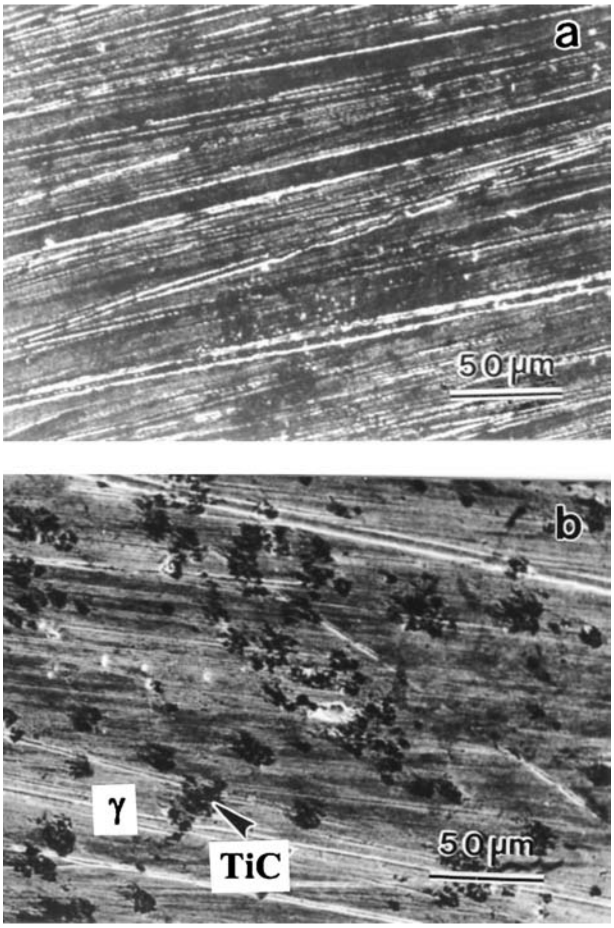

Figure 8 SEM micrographs of the worn surface of (a) Hadfield Mn-steel and (b) composite.

duce the contact area for abrasion. The low coefficient of friction of TiC and high oxidation resistance of matrix due to the presence of Al and Si further contribute in lowering the wear rate. The worn surface of the Hadfield-Mn steel (Fig. 8a) shows long continuous grooves, which form as the SiC particles plow along the surface and remove material along sides. On the other hand, the severity of micro plowing is less in the case ofcomposite with mostly austenitic type matrix and TiC reinforcement (Fig. 8b). TiC particles, exposed to the worn surface, blunted the tips of the abrasive particles due to its high hardness. Hence, depth of the groove is less in the case of composite compared to Hadfield-Mn steel. No particle cracking, pull out or cracking at the particle matrix interface is noticed, suggesting that the bonding between the matrix and the TiC particle is strong.

It is possible to synthesize austenitic steel matrix composite reinforced with TiC particles by aluminothermic reduction of siliceous sand in the presence of C and ferro-manganese. The austenite, stabilized by the addition of Mn, looses its stability at high temperature. On annealing, Fe_2AlMn phase forms depleting the Mn content of the matrix resulting higher amount of ferrite in the matrix of annealed composite compared to the same in as-cast composite. Wear resistance of the as-cast composite under abrasive condition is better than that of the Hadfield Mn-steel.

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