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## Characterization of microstructure and tensile fracture behavior in a novel infiltrated TiC–steel composite

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## ABSTRACT

The tool steel matrix composite was successfully produced by a novel infiltration method so as to have TiC reinforcement particles more than 57 vol.%. The interface between  $\alpha$ -Fe and TiC was found to be semi-coherent nature and the orientation relationship was clearly confirmed, i.e.  $[012]_{\alpha$ -Fe //  $[110]_{TiC}$ ,  $(200)_{\alpha$ -Fe //  $(\overline{2}24)_{TiC}$ . Tensile fractures consisted mainly of {100} microcleavage of TiC grains with no interfacial decohesion. The increased strength of the composite at 700 °C is rationalized in terms of mutually interactive influences of load transfer mechanism and the matrix strengthening resulted from the increased density of dislocations.

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As one of the effective ways to increase the specific strength of materials, the incorporation of ceramic particulate reinforcements into metal matrices becomes more important as a result of its relatively low costs and characteristic isotropic properties. These materials were composed mainly of light metal matrices such as aluminum or magnesium, and reinforced with high strength ceramics particulate such as SiC, B<sub>4</sub>C, Al<sub>2</sub>O<sub>3</sub>, and Al<sub>3</sub>Ti [1–6]. These composites have been extensively investigated. However, little attention has been given to the development of steel matrix composites, although steel has higher stiffness, strength, and cost-competitiveness than Al or Mg [7]. The titanium carbide. TiC is well-known for reinforcement in ferrous steel matrix because of its high melting point, low density and high elastic modulus [8]. Most importantly, the TiC exhibits good melt wettability with iron and steel matrices [9]. Generally, TiC-Fe composites have been produced by powder metallurgy which is an attractive processing route for steel matrix composites because a wide range of reinforcement volume fraction and size can be used and a solid state process produces the sound metal-ceramic interface by avoiding extensive reactions [7,10]. However, the fabrication process of steel matrix composites by this powder metallurgy has several drawbacks such as high cost, inhomogeneity and limited scale-up of components [8]. As efforts for solving these problems, the present work was initiated to study the feasibility of producing of TiC-SKD11 composite by infiltration process. The

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http://dx.doi.org/10.1016/j.scriptamat.2015.09.028 1359-6462/© 2015 Elsevier Ltd. All rights reserved. microstructure and the tensile fracture behaviors of the infiltrated TiC-steel composites have been investigated in order to provide more insight on the very limited information for the process-structure-property relationship.

The investigated cermet is a TiC particulate reinforced SKD11 tool steel matrix composite. For comparison with light metal matrices such as aluminum or magnesium, the steel is simply called as matrix phase although the volume fraction of steel is much lower than 50% in the present study. Ceramic TiC (Changsha Langfeng Metal Materials Co., Ltd. 2500 mesh) powder was compressed under isostatic pressure of 80 MPa, and sintered at 1400 °C for 60 min under argon atmosphere in a graphite furnace. The preform with a dimension of diameter 55 mm and height 25 mm was measured to have 38.3% open porosity by the liquid displacement method in distilled water. The special care was made to have a weak-bonded preform ensuring that a pressurized infiltration of molten steel allows each TiC particle to be separate by debonding. The starting steel matrix material was JIS SKD11 with the chemical composition of 1.4C-11.1Cr-0.80Mo-0.23 V (all in weight percent, equivalent to AISI D2). The SKD11 with 5 kg weight was melted under vacuum at  $10^{-4}$  Torr in a furnace (ALD Vacuum Technologies GmbH, model VIM III-P). Infiltration by molten steel into the TiC preform, which was supported tightly by MgO crucible, was then conducted at 1500 °C for 5 min with argon gas pressure of 0.5 MPa, followed by furnace cooling. No further thermal treatment was performed in the present study.

Uniaxial tensile tests were carried out on an electromechanical actuator driven dynamic machine (Instron Engineering Corporation, model

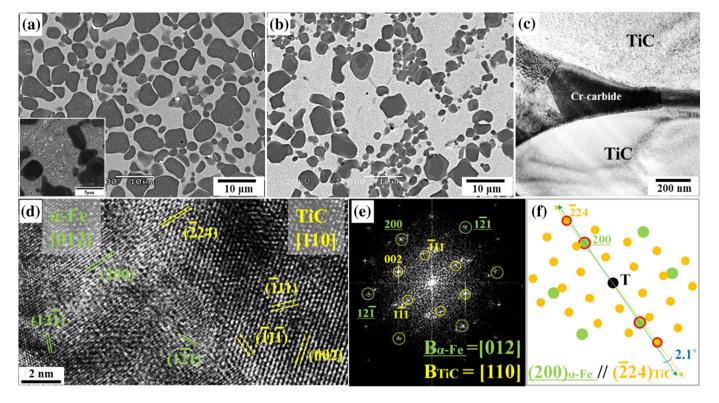






8862) equipped with a 100 kN load cell. The tensile tests were conducted at 25 and 700 °C with a cross head speed of 1.5 mm/min and 0.5 mm/ min, respectively. The specimens have been machined by wireelectrode cutting with a cross-section of  $6.5 \times 1.5 \text{ mm}^2$  and a gauge length of 5 mm. The gauge sections were mechanically polished to minimize the effects of surface irregularities and finish, using emery papers impregnated with silicon carbide from 500 to 2000 grit. The tensile properties (yield strength, ultimate tensile strength, elongation) were obtained based on the average of three tests. The microstructures of the composites were studied using a JEOL JSM-5800 scanning electron microscope (SEM) with a tungsten filament operating at 20 keV. The crystal orientation of the sample was investigated by a FEI Helios D440 field emission scanning electron microscope equipped with a TSL-OIM electron back-scattered diffraction (EBSD) analyzer. Transmission electron microscopy (TEM) characterization was performed on a field-emission JEOL JEM-2100 F microscope operating at 200 keV. The thin foils were prepared by a focused ion beam (FIB) system.

Fig. 1a shows a typical microstructure of the low-pressurizing infiltrated composite, which is composed of a reinforcement of TiC grains, with an average TiC grain size of ~4 µm. For comparison, the 45% volume fraction of TiC reinforced SKD11 composite was produced by conventional powder metallurgy and its microstructure is presented in Fig. 1b. The infiltrated TiC-SKD11 composite showed a more homogeneous distribution of TiC particles with very low frequency of TiC contiguity. This characteristic of microstructure is expected to ensure isotropic properties and uniform distribution of stresses into the composite. As shown in Fig. 1c, the infiltration of SKD11 molten steel was complete so that the penetration of the liquid metal reached geometrically complex regions. The TiC particles have completely wet by the SKD11 steel matrix. Some Cr-rich carbides can be found to bridge TiC particulates. The steel binder phase shows a martensitic structure subjected to tempering by slow cooling after infiltration, as shown in the inlet of Fig. 1a. According to Naidich [11], the contact angle  $(\theta)$  was so as low as 28° at 1490 °C under vacuum that good wettability of TiC by iron alloys can be achieved. Furthermore, Terry and Chinyamakobyu studied in situ production of Fe-TiC composites by reactions in liquid iron alloys. They reported that the use of low carbon alloy favors dissolutions of the TiC, which leads to a good wetting of the TiC by the liquid alloy. Nevertheless, Kaplan et al. [12], who fabricated TiC-1080 steel cermets by the pressureless-infiltration, reported that small flaws were frequently found at the metal-ceramic interfaces, and quasi-static failure occurred by extension and blunting of these interfacial flaws. However, we could not find any interfacial flaws at the TiC-matrix interface in the present study. Fig. 1d presents the high resolution (HR) TEM image of the TiC-steel matrix interface. The interface between the TiC and steel matrix is found to be faceted when it is viewed down to the atomic scale. The faceted interfaces correspond to the dense atomic planes of TiC, such as  $(\overline{1}1\overline{1})$ , (002) planes. The orientation relationship between  $\alpha$ -Fe and TiC for this interface is clearly confirmed, i.e.  $[012]_{\alpha-\text{Fe}} // [110]_{\text{TiC}}$ ,  $(200)_{\alpha-\text{Fe}} // (\overline{2}24)_{\text{TiC}}$ , according to the interface diffraction pattern produced by Fast Fourier Transform (FFT) analysis (Fig. 1e). However, the analysis of computed spot patterns (Fig. 1f) and HR-lattice image suggested that  $(200)_{\alpha\text{-}Fe}$  is slightly deviated 2.1° from  $(\overline{2}24)_{Tic}$ . The TiC-steel matrix interface is likely to have elastic misfit strain due to its semi-coherent nature. Cha et al. [13] measured the residual misfit of the interface between Fe and TiC precipitates localized along TiB<sub>2</sub> basal plane. Its value was 1.85%, which was accommodated by an array of disconnections whose Burgers vector promoted a rotation of planes, and thus a change in the orientation relationship. It is interesting to realize that although steel matrix was formed by solidification process, the TiC and the steel matrix make semi-coherent interface containing the orientation relationship. Terry and Chinyamakobvu [14] showed that the TiC would be expected to dissolve if the proportions added (C, Ti) are below the solubility limits of TiC in pure molten iron. Additionally, according to pseudo-binary



**Fig. 1.** SEM micrographs of TiC–SKD11 composites produced by (a) the infiltration in the present study and (b) the powder metallurgy for comparison. (c) TEM micrograph of the infiltrated composite revealing TiC particles completely wet by the SKD11 steel matrix. (d) HR-TEM image of the interface between TiC and steel matrix in the infiltrated composite shown in Fig. 1c. The zone axis is  $[012]_{cr-Fe} //[110]_{TiC}$ . Note that the faceted semi-coherent interfaces terminate on the dense atomic planes of TiC. (e) FFTed and (f) computed diffraction patterns of the interface showing that  $(200)_{cr-Fe}$  is slightly deviated 2.1° from  $(\overline{2}24)_{TiC}$ .

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