Communications

In Situ Formed (TiW)C Phase in Iron Matrix

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The TiC ceramic phase is ideal particulate reinforcements in steel and iron matrix composites. In fact, as early as the 1950s, TiC/Fe composites were studied[1] and have been applied to cutting, machining, and wear resistance. They have been produced through powder metallurgy. However, the process has intrinsic limitations for production costs and material qualities. Solidification processing has appeared to be one of the most economical and versatile techniques for producing metal matrix composites (MMCs) and has been widely used in the production of light MMCs such as Al, Mg, *etc*. In contrast, because they have high melting points, relatively little work had been conducted on the solidification processing of iron matrix composites until the *in situ* synthesis technique in melts was developed recently.

For the last decade, much attention has been paid to the *in situ* synthesis technique of the MMCs. Its eminent advantage is that it eliminates interface incompatibility of matrices with reinforcements by creating more thermodynamically stable reinforcements based on their nucleation and growth from the parent matrix phase. Although it was developed first for preparation of aluminum matrix composites, the technique has also been applied to synthesize iron matrix composites, mainly TiC/Fe.[2–6] Spherulitic and uniformly distributed TiC reinforcements could successfully be synthesized in iron matrices. This opened a door for solidification processing of iron matrix composites. However, it should be noted that there is great difference in density between TiC and iron melt. Relatively light TiC particulates are formed first and tend to float up during solidification, resulting in their segregation in cast composites. (TiW)C phase may be a more prospective candidate for reinforcements synthesized *in situ* in iron melt, because its density is a little higher than that of TiC and approaches that of iron melt.

The present work investigated the feasibility of *in situ* synthesis of the (TiW)C phase in iron matrix. It is expected that the preliminary results can be significant in promoting the development of *in situ* synthesized iron matrix composites.

Commercial grade of powders of titanium, tungsten, graphite, and iron were used for the preparation of batch. The batch, Fe-8 wt pct Ti-9.4 wt pct W-2.6 wt pct C, was prepared to obtain 20 wt pct (TiW)C-Fe composites. After sufficient mixing, the powders were uniaxially compacted in a die of 25 mm in diameter under a pressure of 270 MPa. A sample weighing 10 g was heated in an electric arc furnace using a nonconsumable W electrode. In order to achieve a homogeneous microstructure, melting was done 2 times. After the first time melting, the sample was turned upside down and melted again. The reaction product was weighed, cut, ground, and polished for metallographic observation. Scanning electron microscopy (SEM) in combination with energy-dispersive X-ray analysis (EDAX) was used to examine microstructure and to perform compositional microanalysis. In order to identify the nature of the formed phases, X-ray diffraction analyses with Cu K_α radiation were done on the sample.

Figure 1 shows the X-ray diffraction pattern. Besides iron peaks, there are also prominent peaks of TiC and/or (TiW)C in Fe-W-Ti-C material. Those of TiC and (TiW)C phases exactly overlap and no distinction can be made between them. So, the formed carbides need to be identified further by microstructural observation and microanalysis with SEM and EDAX. Furthermore, it is noticed that in the iron matrix surplus carbides such as cemetite (Fe_3C) seem to be avoided.

Figure 2 is the microstructure of Fe-Ti-W-C material. It can be seen that the formed carbides are spherulitic and uniformly distributed in the matrix, which makes them appropriate as reinforcements in iron matrix composites. Energy-dispersive X-ray analysis was conducted on the carbide, as shown in Figure 3. It is shown that the *in situ* formed carbide is (TiW)C phase, rather than TiC phase.

Numerous microstructural observations indicate that many (TiW)C particulates contain a dark core. Figure 3 shows such characteristic feature of the (TiW)C phase. On this fine scale microstructure, furthermore, it is seen that there are several distinct outlines around the core, which indicates that the distribution of the elements within the phase is probably nonuniform. The line scanning across the phase, shown in Figure 3, was conducted with EDAX, and the result is shown in Figure 4. Evidently, the phase substantially consists of titanium, tungsten, and carbon atoms, and does not contain iron. But, on a close examination, it is found that titanium and tungsten atoms indeed are unevenly distributed. In the dark core are the most titanium and least tungsten atoms, while beyond it, the content of tungsten increases and that of titanium decreases. From the core outward, there are positive and negative gradient distributions of titanium and tungsten atoms, respectively. The outlines seem to be traces of the solidification front of the phase. Due to the limitation of resolution of EDAX, it failed to determine the exact composition of the core. But, it is reasonable to deduce the rich core, probably TiC phase, was formed first in iron melt, and acted as the solidifying nucleus of the (TiW)C.

As an electric arc furnace characterizes rapid heating and cooling in synthesizing materials, microstructural constituents in a synthesized product almost all result from reactions in liquid melt. During heating the batch, iron first may melt, its melting point being the lowest among the constituents. Then, the others may dissolve the iron melt, forming a homogenous melt. During subsequent cooling, the TiC phase first forms through a direct reaction between solute Ti and C in the melt. This is confirmed by the titanium-rich core of the (TiW)C (Figures 3 and 4). Due to a lack of phase diagram of the Fe-Ti-W-C system, it is impossible to determine the solubility of the alloying elements in liquid iron. From an isothermal section of the phase diagram Fe-Ti-C at 1550 °C (Figure 5), it can be seen qualitatively that the TiC has a relatively low solubility in liquid iron.

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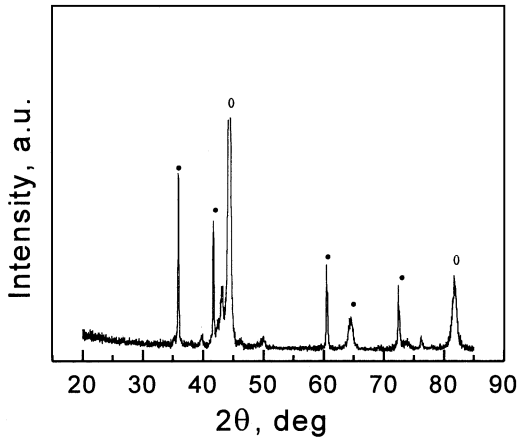

Fig. 1—X-ray diffraction pattern of Fe-Ti-W-C material: (o) Fe and (•) TiC/(TiW)C.

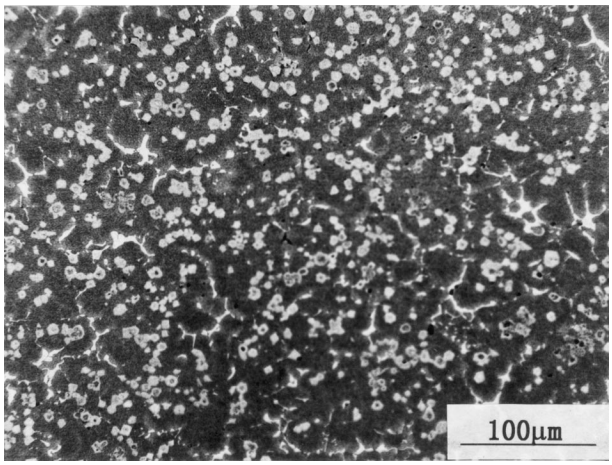

Fig. 2—Backscattered electron showing the microstructure of Fe-Ti-W-C material.

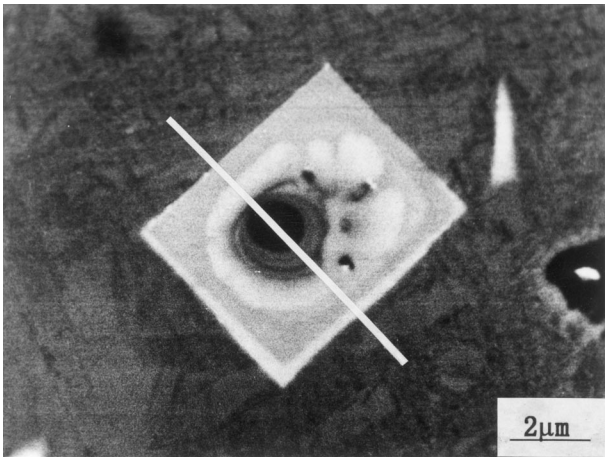

Fig. 3—*In situ* formed (TiW)C phase; the line indicates the position of line scanning.

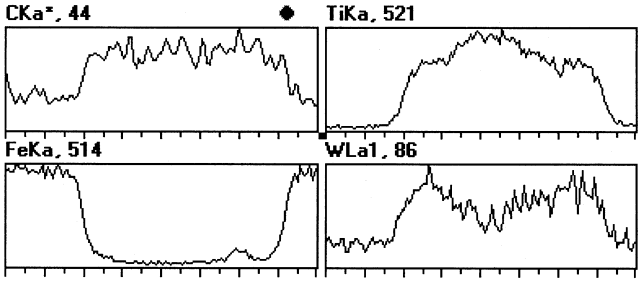

Fig. 4—EDAX spectrum of the (TiW)C phase, as shown in Fig. 3.

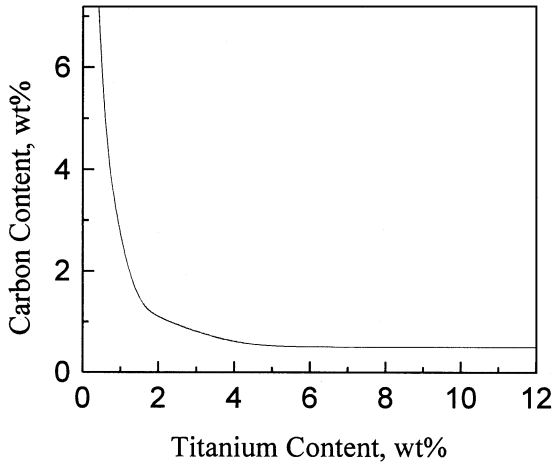

Fig. 5—An isothermal section of the phase diagram Fe-Ti-C at 1500 °C.[7]

Therefore, a profuse precipitation of the TiC must have occurred in the melt during cooling. With temperature dropping further, the solubility of tungsten in the melt decreased and tungsten atoms were expelled from the melt. They entered into the growing TiC phase by substituting titanium ones. This was confirmed by the tungsten-rich periphery of the phase (Figures 3 and 4). Consequently, the formation of the resultant (TiW)C phase is closely related to solubility change of the alloying elements in the melt during cooling.

As is well known, the TiC phase is indeed a very effectively heterogeneous nucleus of metal and alloy melts and has been widely used to refine solidifying microstructures of castings. Recently, the present authors found that the TiC phase is also an active solidifying nucleus of $(Fe, Mn)_3C$ carbide in TiC/Hadfield steel composites.[6] Evidently, such an ability of the TiC phase would favor refinement of reinforcements in iron matrix composites. Undoubtedly, in this work, perfect morphology and distribution of the (TiW)C phase in iron matrix should be attributed to the heterogeneous nucleation of the TiC phase.

At present, since the chemical constitution of the synthesized (TiW)C phase is unknown, the exact calculation of its density is impossible. Here, a rough evaluation is made. In the phase, an atomic ratio of titanium and tungsten is assumed to be 1:1, and thereby, its formula is $(Ti_{0.5}W_{0.5})C$. Considering that the (TiW)C has the same crystal lattice and lattice constant as TiC, the density of the $(Ti_{0.5}W_{0.5})C$ is calculated. The result shows that it is 9.07 g/cm^3, while those of TiC and iron are 4.25 and 7.8 g/cm^3, respectively. This indicates that the $(Ti_{0.5}W_{0.5})C$ does have a density closer to iron than TiC.

The present work indicates that (TiW)C could be synthesized in iron matrix. The (TiW)C phase is spherulitic and uniformly distributed. Within the (TiW)C phase, however,

there is uneven distribution of elements. The nearer the core of the phase, the higher the content of titanium and the lower the content of tungsten. The titanium-rich core must be a solidifying nucleus of the (TiW)C phase. The (TiW)C phase, having a density matchable for iron melts, seems to be more appropriate as reinforcements synthesized *in situ* in iron melts for large-scale composite ingots, where they take a much longer time to solidify. Undoubtedly, more comprehensive research work needs to be done before such reinforcements are to be put into commercial use.

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A Novel Approach for Optimizing the Fracture Toughness of Precipitation-Hardenable Al-SiCp Composites

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Several studies have investigated the effect of microstructural variables (volume fraction, size, shape, distribution of particles, matrix microstructure, and interface composition) on the fracture toughness of discontinuously reinforced aluminum (DRA) composites.[1,2,3] Recently, it was shown that the fracture toughness of the DRA composites may also be improved by altering the matrix grain and subgrain structures.[4] Dutta *et al.*[4] utilized the mechanism of particle stimulated nucleation (PSN) of recrystallization during deformation processing to refine the matrix microstructure, and demonstrated that this resulted in a significant enhancement of fracture toughness *and* strength in the peak-aged (-T6) state. However, it was noted[4] that once the reinforcement distribution has been homogenized, and the matrix grain structure refined, by deformation processing to large total strains in the PSN regime, further improvement in the fracture toughness is not achievable without leaving the matrix in the underged (UA), and therefore, unstable state. Hence, a novel heat treatment approach is necessary in order to improve the fracture toughness of DRA beyond that obtainable by processing to a large total strain in the PSN regime, followed by peak aging.

In DRA composites, damage initiation usually begins by cracking of particles[5,6] or by microvoid nucleation and growth within the matrix between particle clusters.[7] For high strength matrixes (*e.g.*, -T6) displaying low work-hardening rates, this damage is followed by a high degree of strain localization because of the inability of the matrix to work harden, leading to immediate fracture. For relatively soft matrixes, which can work harden appreciably (*e.g.*, UA or naturally aged condition), on the other hand, strain localization is limited, and significant deformation can occur following the initial particle fracture event, before eventual failure. An overaged (OA) matrix can work harden, and is therefore susceptible to less strain localization, but displays low fracture toughness due to premature microvoid nucleation at coarse, incoherent precipitates, which usually form adjacent to the particle/matrix interface.[8] The ideal microstructure for optimal strength-fracture toughness combination, therefore, is one which allows loading of the reinforcement particles to failure, but subsequently inhibits crack transition into the matrix by allowing it to work harden significantly.

Sugimura *et al.*[9,10] have shown that when a crack in a plastically weak solid (low yield stress, σ_{ys}, and low work-hardening exponent, n) approaches an interface with a plastically hard solid (high σ_{ys} and high n), the crack tip is shielded from the remote load (*i.e.*, there is reduction in crack tip stress field). This inhibits transition of the crack from the plastically weaker to the plastically stronger solid. Based on this observation, it is envisioned that if a soft, precipitate-free zone (PFZ) is present in the matrix immediately adjacent to each reinforcement particle, and this is surrounded by a hard, work-hardening matrix (*e.g.*, in a slightly overaged state), the cracks from the failed particulates will easily transition into the PFZ, but will be inhibited from transitioning readily from the soft PFZ into the surrounding hard matrix. Meanwhile, following the initial particle fracture event, the matrix will work harden, and thereby prevent rapid strain localization and fracture. This conceptual sequence of events (schematically shown in Figure 1) should allow significant matrix plasticity (and therefore yield increased fracture toughness), before separate microvoid nucleation events occur in the hard matrix regions, resulting in eventual failure.

The formation of PFZs has been reported in Al-based composites at both grain boundaries and interfaces.[11] In general, grain boundary PFZs are detrimental to the mechanical properties of Al alloys,[12,13] although in composites, they seem to have little effect.[14] The PFZs at the particle/matrix interface, in conjunction with an overaged matrix, on the other hand, have been noted to enhance ductility by as much as a factor of 4, albeit with a substantial drop in tensile strength.[11] Whereas PFZs next to grain boundaries are associated with the formation of coarse boundary precipitates, interfacial PFZs in composites are thought to occur due to

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