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Effects of cooling rate and heat treatment on the microstructure of iron-based titanium carbide composites

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The microstructural response of iron–carbon–TiC components produced by a liquid route to changes in cooling conditions and post-solidification heat treatments has been investigated. Heat treatments of pure Fe–TiC composite materials have been found to produce only minor changes in the TiC morphology and distribution with reduction in hardness resulting from some TiC precipitation and depletion of the iron matrix of titanium and carbon. As-cast composites containing Fe–2.4% C/10% TiC and Fe–3.27% C/10% TiC exhibit a white cast iron matrix containing dispersed TiC. Subsequent heat treatment of this material at 750 °C renders the matrix malleable, replacing the cementite by free graphite with no apparent alteration to the TiC morphology or distribution.

1. Introduction

Previous investigations [1, 2] by the present authors examined the feasibility of producing iron-based titanium carbide composites by liquid-based routes, either by the direct addition of TiC powders to liquid iron alloys or by the production of TiC *in situ* in the liquid iron alloy. The viability of such routes is dependent upon the identification of conditions where TiC is readily wetted by the liquid iron alloy allowing dispersions of TiC within that alloy to be maintained.

The present authors [3] have found that such conditions can be established when TiC is in contact with iron with a high carbon content, and/or high carbon and high titanium contents. The solidification of such materials essentially produces a dispersion of TiC in a white cast iron matrix. While such a composite material will undoubtedly provide an extremely hard surface, its use is likely to be limited by the brittle nature of the white cast iron matrix and the resultant lack of toughness in the composite.

When molten, low-carbon iron alloys are contacted with titanium carbide powders, substantial dissolution of the titanium carbide occurs. The subsequent solidification of such a material is then likely to yield an iron alloy matrix supersaturated with respect of titanium carbide. Subsequent heat treatments may then permit precipitation of titanium carbide with a resultant alteration to the material's mechanical properties. It is therefore necessary here to study the response of both pure iron/titanium carbide composites and high-carbon iron/titanium carbide composites to heat treatments.

The purpose of the present investigation was to examine the effects of cooling conditions and post-solidification heat treatments on the microstructure and properties of iron-based titanium carbide composites produced by a liquid route. It has been our aim to promote conditions whereby the matrix microstructure can be modified to achieve a combination of wear resistance and toughness in the final composite. Hardness measurements have been used initially as a simple means of assessing the effect of heat-treatment procedures on mechanical properties.

2. Experimental procedure

In the majority of the experiments, induction heating of the relevant iron alloy, mixed with TiC powder, was used to form 50 g lumps of composite material. The equipment, procedure and raw materials used have been described in detail in our previous investigations [1–3]. A holding time of 10 min at 1600 °C was applied to the composite material in each case.

After solidification and cooling, the 50 g lumps were cut into smaller sections of 5–10 g for heat treatment, optical examination and hardness measurements.

Samples for heat treatment were encapsulated in argon-filled silica tubes before being placed in a muffle furnace set at the required temperature. After the desired period the samples were removed from the furnace, left to cool in air and then removed from the silica tubes. They were then prepared for metallographic examination and X-ray diffraction analysis. Hardness measurements were carried out on a Vickers machine with a diamond indentor using a 10 kg load.

TiC powders of various particle size, were used in the experiments.

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3. Results and discussion

3.1. Pure Fe–TiC composites

Table I summarizes the experiments performed and Fig. 1 shows some typical microstructures of unetched specimens.

Run R17 was a control experiment in which pure iron was melted under the same conditions as all the other experiments. The resultant microstructure was seen to be single phase alpha with rugged grains and a very variable grain size. As expected, where there are no alloying additions, the hardness value was very low, 65 H_v with a 10 kg load.

Runs R13 and R14 involved the addition of low-volume fractions of TiC to pure iron melts, followed by cooling under flowing argon. The resultant microstructures, e.g. Fig. 1a, showed fine spherical precipitates of 1/2–1 μm diameter with long, thin platelets or whiskers, the longest of which were up to 20 μm long. Although the microstructures were virtually the same, R13 had a hardness value of 226 H_v and R14 178 H_v; the difference of 48 H_v seems rather large to be accounted for solely by the small difference in weight per cent TiC added (4.03 compared with 3.36) and it is therefore suspected that R13 was cooled somewhat faster than R14.

In run R18, a low fraction of TiC (3.21 wt % TiC) was added once again to pure iron but in this case the composite was quenched in water within 1 min of

TABLE I Hardness response of pure Fe + TiC composites to heat treatment

Run	Matrix alloy	TiC filler (wt %)	As-cast hardness (H_v)	Hardness (H_v) after 48 h at 1200 °C	Hardness (H_v) after 48 h at 1000 °C	Hardness (H_v) after 48 h at 800 °C
R17	Pure iron	–	65	–	–	–
R18[a]	Pure iron	3.21	379	198	145	178
R14	Pure iron	3.36	178	–	–	–
R13	Pure iron	4.03	226	226	147	147
R16	Pure iron	11.06	283	272	186	204

[a] Water quenched soon after solidification.

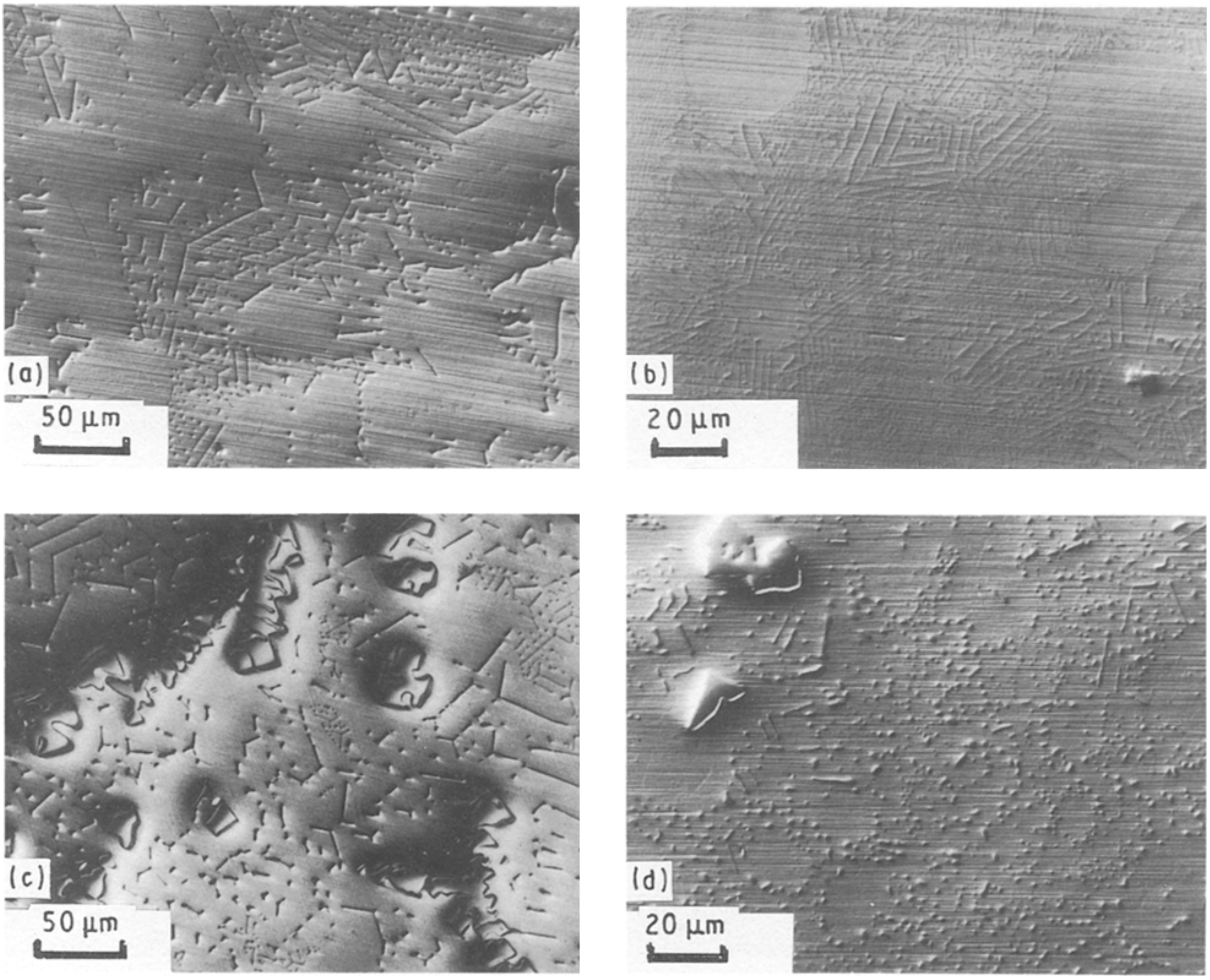

Figure 1 TiC dispersions in pure iron after induction melting. (a) Fe–4.03 wt % TiC air cooled from 1600 °C, (b) Fe–3.21 wt % TiC water quenched from 1600 °C, (c) Fe–11.16 wt % TiC air cooled from 1600 °C, (d) Fe–3.21 wt % TiC treated as in (b) then heat treated under argon for 48 h at 1200 °C.

switching the induction heating power off. The microstructure observed (Fig. 1b) consisted of very fine precipitates, both spherulites and elongated platelets, of TiC. In each case these precipitates were much smaller than those observed in runs R13 and R14. The hardness value was accordingly much higher at 379 H_v than that for R13 which was 226 H_v.

In run R16 a much larger addition of TiC (11.66 wt %) was employed. It was observed in this case that some of the added TiC floated to the top of the melt and formed a crust. The microstructure produced after cooling consisted of a background of fine spherical and platelet precipitates interspersed with a larger particle size dispersion (Fig. 2c). The large dispersed particles had an average size of 8 µm, comparable to the original particle size of the TiC used. The morphology of the majority of the smaller dispersed second-phase particles suggests a tendency for interdendritic segregation. The hardness of the specimen was measured and found to be 283 H_v.

It can be concluded that faster cooling rates and increased additions of TiC give greater hardness values. Dissolution and reprecipitation of TiC is favoured in pure iron and any excess or undissolved TiC will disperse in the resultant liquid alloy. This is indicated by R16.

Heat-treating specimens from R13 alternately at 1200, 1000 and 800 °C produced very little noticeable microstructural change. There was slight evidence of spheroidization of the TiC in the heat treatments at 1000 and 800 °C. The heat treatment at 1200 °C produced no change in the specimen hardness but heat treatments as both 1000 and 800 °C reduced the hardness from 226 H_v to 147 H_v. The solubility of both titanium and carbon in iron is much higher at 1200 °C than at 1000 and 800 °C.

It is assumed that migration of titanium and carbon during the 1000 and 800 °C treatments precipitated TiC either independently or on existing TiC particles. The resultant depletion of the matrix of the titanium and carbon solutes is thought to be responsible for observed drops in hardness.

In addition to this, the heat treatments are likely to relieve any residual stresses produced in the material during the solidification process. The absence of any reduction in hardness during the 1200 °C heat treatment would, however, suggest that these effects have a negligible influence on the hardness of the material.

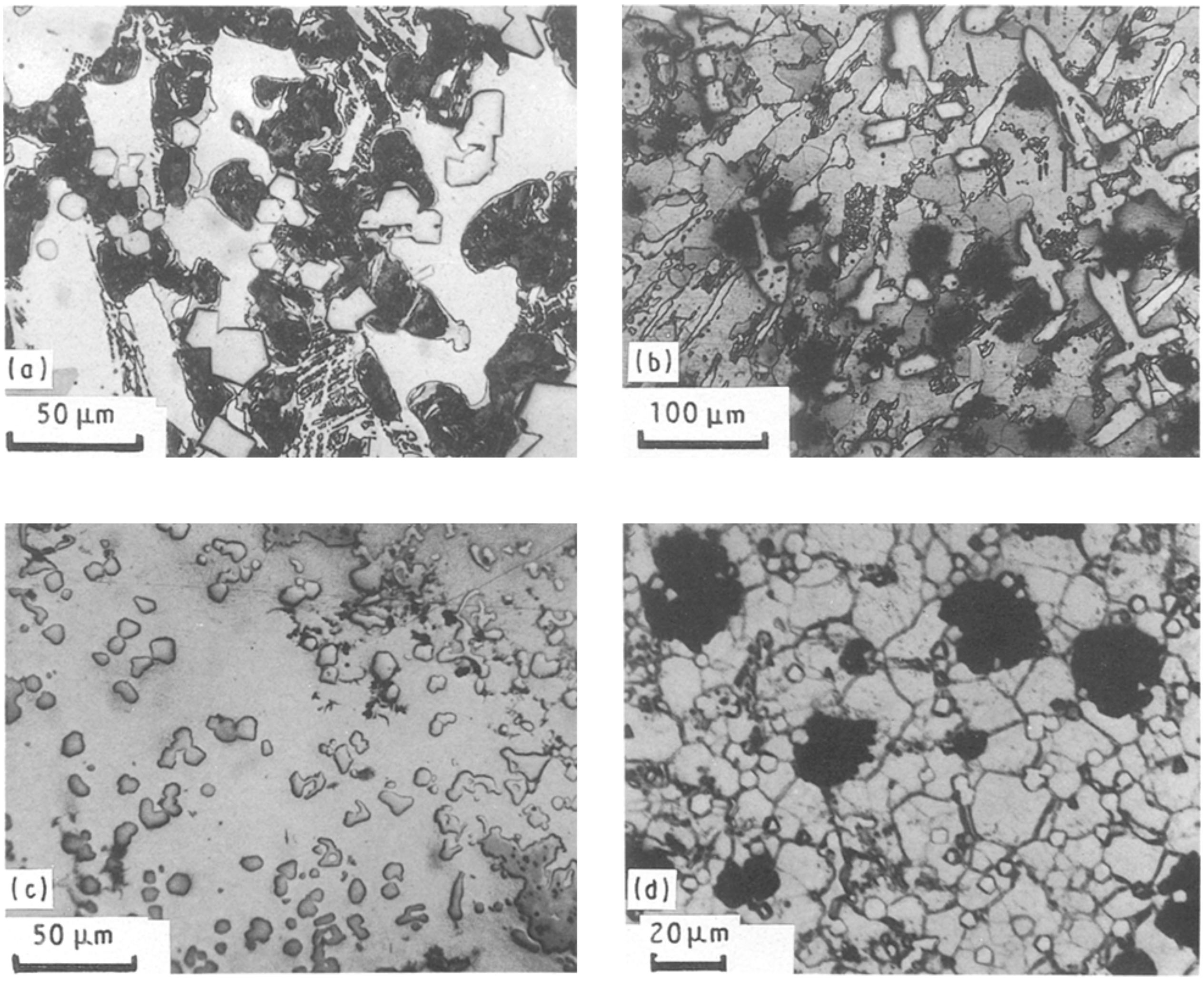

Figure 2 Cast iron structures with TiC dispersions. (a) Fe–2.3 wt % C alloy with 10 wt % TiC. (b) Alloy in (a) heat treated at 750 °C for 72 h. (c) Fe–3.24 wt % C alloy with 10 wt % TiC. A better dispersion of TiC was observed as a result of good mixing due to levitation. (d) Alloy in (c) heat treated at 750 °C for 72 h.

Heat treating specimens from R16 did not produce any noticeable microstructural changes either. The 1200 °C heat treatment once again produced only a small decrease in hardness. The 800 °C heat treatment, however, produced a smaller decrease in the hardness than did the 1000 °C treatment for the same period of time (48 h). It is believed that because the diffusion rates of titanium and carbon in iron at 800 °C are slower than at 1000 °C, the solute depletion of the matrix at the lower temperature was less than at the higher. It would be anticipated that if the heat treatment at 800 °C was continued for a longer period of time, a hardness value lower than that of the 1000 °C heat treatment would eventually result.

The heat treatment of specimens from run R18 produced the most dramatic microstructural changes. In this case precipitation had been suppressed during solidification by the fast cooling rate. Subsequent heat treatments permitted more extensive TiC precipitation.

As expected, the sample heat-treated at 1200 °C exhibited the greatest hardness value consistent with higher levels of titanium and carbon remaining in solution in the matrix. The heat treatments at 1000 and 800 °C produced a similar trend in hardness values to those already discussed for R16.

3.2. Higher carbon iron–TiC composites

Table II summarises the experiments conducted with high carbon iron–TiC composites. Fig. 2 illustrates representative microstructures obtained.

In run R6 a medium carbon (2.3 wt %) iron was melted with about 10 wt % TiC of 30–70 μm size. Owing to insufficient agitation, some TiC remained floating on top of the iron melt during the operation and consequently the fraction of TiC retained in the composite was less than 10 wt %. The microstructure obtained from the solidification of this material showed massive eutectic cementite which formed on cooling and coarse pearlite which formed as a result of the austenite decomposition (Fig. 2a). The TiC was dispersed randomly in both phases.

Heat treatment of R6 at 750 °C yielded well-defined ferrite grains and a greatly reduced pearlite phase (Fig. 2b). The eutectic cementite was reduced in size to about 40 μm and graphite precipitation occurred both as a flake network round grain boundaries and as separately nucleated nodules. There was little apparent change in the particle size of the dispersed TiC phase.

In experiment 3M, a sample of about 1 g Fe–3.24% C was levitation-melted with the addition of 10 wt % TiC powder of 5 μm size. In this case the improved mixing conditions led to a better dispersion of TiC; the microstructure obtained was a fine dispersion of TiC in the iron matrix (Fig. 2c). Heat treatment of this sample at 750 °C yielded a slightly reduced TiC particle size and the precipitation of graphite as shown in Fig. 2d. The resultant microstructure is essentially that of a malleable cast iron structure with TiC dispersed in it.

The microstructural responses of the high-carbon iron–TiC composites to heat treatments can best be understood by considering briefly the metallurgy of cast irons. These are primarily alloys of iron which contain more than 2 wt % carbon and between 1 and 3 wt % Si [4, 5]. There are four basic types of cast iron: white, grey, ductile and malleable iron. White cast iron is formed when the carbon in solution in the molten iron does not form graphite on solidification but combines with iron to form the metastable phase cementite. Fig. 2a (run R6) shows a typical white cast iron structure with TiC dispersed in it. The white cast iron matrix comprises large particles of cementite (white) and coarse pearlite, the pearlite being the product of the decomposition of both primary and eutectic austenite. The formation of white cast iron is favoured by low carbon and low silicon contents and rapid solidification. Higher carbon and silicon contents and slower cooling rates favour the formation of a grey cast iron where a substantial proportion of the carbon content separates as flake graphite. Additions of magnesium or cerium have been found radically to alter the morphology of this flake graphite, producing a nodular or spherical graphite (s.v.) cast iron. As the cementite formed in white cast irons is detrimental to mechanical properties, in particular toughness, heat treatments can be applied to impart ductility to an otherwise exceedingly brittle material. Thus malleable cast irons are traditionally produced by a two-step heat treatment. Such treatments involve first heating at 925 °C to allow the combined carbon to dissolve in austenite and then to migrate to preferred sites and precipitate there as irregular nodules of graphite known as temper carbon. The second heat-treatment

TABLE II Heat-treatment response of high C–Fe + TiC composites

Run	Experiment	Phases identified (XRD)	Comments
R6	Induction melting of Fe–2.3 wt % C alloy (1590 °C) with 10 wt % TiC	Fe, Fe_3C, TiC	Induction melting does not give as good mixing as levitation, so there was non-uniform TiC phase distribution.
R6HT	Heat treatment of R6 at 750 °C for 72 h.	Fe, C_{gr}, TiC Fe_3C (weak)	Fe_3C decomposed and C_{gr} was precipitated.
3M	Levitation of Fe–3.24 wt % C alloy (1550 °C) with 10 wt % TiC	Fe, Fe_3C, TiC	Very good TiC dispersion.
3MHT	Heat treatment of 3M at 750 °C for 72 h	Fe, C_{gr}, TiC	Malleable cast iron microstructure obtained.

step involves a slow furnace cooling, particularly through the eutectoid temperature, in order to allow most of the carbon remaining in solution in the austenite to precipitate on the existing temper carbon nodules.

In experiment 3MWT in the present study, the heat-treated sample Fe–3.24% C + 10% TiC was held at 750 °C, just above the eutectoid temperature, for 72 h and the same effect was achieved. The resulting microstructure (Fig. 2d) shows TiC dispersed in a typically malleable iron microstructure. Heat treatment of the lower carbon content sample, Fe–1.4% C + 10% TiC at 750 °C for 72 h produced the microstructure shown in Fig. 2b. In this case, while free graphite has been produced, a small amount of cementite is still present in the specimen.

It appears that the microstructural changes which take place during the heat treatment of the various cast irons studied barely change the distribution of the TiC phase. This has interesting prospects as it would appear that incorporation of TiC into cast irons is likely to increase their wear resistance while subsequent heat-treatment procedures will permit essentially independent modification of the matrix microstructure and properties. Work is now in progress on an investigation of the solidification and heat-treatment procedures and alloying additions required to establish appropriate conditions for the production of white, grey, malleable and spheroidal graphite cast irons bearing TiC filler particles. Simultaneous wear and mechanical property testing of the materials produced will allow assessment of optimum processing conditions for designated property requirements. As it has proved possible to disperse TiC in liquid iron, the existing casting technology established for cast iron processing is likely to be applicable for the production and processing of such materials.

4. Conclusions

1. As-cast Fe–TiC composites contain a mixture of fine spherical particles and flake-like particles of TiC.

2. Increased cooling rates reduce significantly the size of both of these forms of TiC and increase the hardness of the material produced.

3. Subsequent heat treatments at 800, 1000 and 1200 °C produce very little microstructural change apart from slight speroidization of the TiC.

4. The noted reductions in hardness values for heat-treated samples can be explained by depletion of the iron matrix of titanium and carbon by TiC precipitation during the heat treatment.

5. Solidification of Fe–2.4% C/10% Ti and Fe–3.24%/10% TiC produces TiC dispersed in a white cast iron matrix.

6. Subsequent heat treatment at 750 °C produces a malleable effect whereby the cementite of the white cast iron matrix is replaced by nodules of graphite. Little or no alteration of the TiC morphology or distribution occurs during this treatment.

7. The possibility exists that by an appropriate choice of alloying additions and processing conditions, a TiC dispersion can be produced in white, grey, malleable or spheroidal graphite cast iron.

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References

1. B. S. TERRY and O. S. CHINYAMAKOBVU, *J. Mater. Sci. Lett.* **10** (1991) 628.
2. *Idem, Mater. Sci. Technol.* **7** (1991) 648.
3. *Idem, ibid.*, in press.
4. I. MINKOFF, "The Physical Metallurgy of Cast Iron" (Wiley, New York, 1983).
5. ASM, Metals Handbook, 9th Edn, Vol. 1 (American Society for Metals, Metals Park, Ohio, 1983) pp. 3–24.

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