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The microstructure and mechanical properties of TiC and TiB_2-reinforced cast metal matrix composites

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TiC and TiB_2 particles have been spontaneously incorporated into commercial purity aluminum melts through the use of a K-Al-F-based liquid flux that removes the oxide layer from the surface of the melt. The combination of spontaneous particle entry and close crystal structure matching in the Al-TiB_2 and Al-TiC systems, results in low particle-solid interfacial energies and the generation of good spatial distributions of the reinforcing phase in the solidified composite castings. The reinforcement distribution is largely insensitive to the cooling rate of the melt and the majority of the particles are located within the grains. Modulus increases after TiC and TiB_2 particle additions are greater than those for Al_2O_3 and SiC. It is thought that interfacial bonding is enhanced in the TiC and TiB_2 systems due to wetting of the reinforcement by the liquid and particle engulfment into the solid phase. TiC-reinforced composites exhibit higher stiffnesses and ductilities than TiB_2-reinforced composites. This has been attributed to stronger interfacial bonding in the Al-TiC system, due to the increased tendency for nucleation of solid on the particle surfaces. *© 1999 Kluwer Academic Publishers*

1. Introduction

1.1. Manufacture of metal matrix composites

There are several techniques available for making metal matrix composites (MMCs) ranging from powder metallurgy methods to casting. One of the most economically attractive is the cast route which employs near standard foundry practice. This inexpensive process, when coupled with a small percentage increase in material cost, makes it a competitive approach for certain lightweight structural and wear-resistant parts.

Problems such as the incorporation of particles into the melt and, in particular, the generation of particle clusters during solidification processing, present significant obstacles to generating a wide range of high quality cast MMCs with properties to rival their powder metallurgy counterparts. For most applications, a homogeneous distribution of the reinforcing phase is desirable in order to maximize the mechanical properties. To achieve good particle dispersions, the mechanisms preventing particles from being introduced into the melt and causing them to cluster once in the melt, need to be understood and overcome.

1.2. Particle incorporation

The obstacles to particle incorporation into the liquid melt are essentially two-fold. There are mechanical barriers, such as surface oxide films, and thermodynamic barriers, often referred to in terms of the wettability. Mechanical barriers can be significantly reduced by the use of fluxes, gas shields or by processing in a vacuum. Overcoming thermodynamic barriers is more difficult. Most ceramic reinforcements are considered to be non-wetting with respect to liquid aluminum at temperatures below 900 °C especially when melt surface oxide films are present. Wettability is often defined in terms of a contact angle less than 90° for a liquid droplet on a ceramic substrate as determined by the sessile drop test [1–3].

To overcome thermodynamic barriers, and to some extent mechanical obstacles to particle entry, melts are often vigorously stirred, the aim being to create a vortex whilst the reinforcement is added to, or below, the melt surface. It is assumed that once non-wetting particles have been introduced into the melt by this method, the high shear induced by stirring affects wetting by a mechanical action and thereby the particles are retained in the melt after solidification takes place [4]. This technique is not without its drawbacks since the prolonged, vigorous stirring also enables oxide skins, gasses and contaminants to be entrained in the melt and 100% transfer of particles to the melt, or their retention in the solidified matrix, is not guaranteed.

It has been shown that by using a flux-casting method, several transition metal carbides and borides can be incorporated into molten aluminum without stirring [5, 6].

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1.3. Particle redistribution

If the ductility of the composite is to be maximized, particle clustering must be avoided so that regions of high triaxial constraint, which promote premature void formation and failure, are not formed. To prevent particles clustering together as they collide in the liquid, the particle–liquid interfacial energy should be low [7], that is, the liquid should wet the particles. Poorly wetting particles will, therefore, have a higher tendency to agglomerate and this can be aggravated by the presence of gas bubbles introduced into the melt by vigorous stirring, since the particles will tend to attach and cluster at the lower energy liquid–vapour interfaces.

To achieve a homogeneous distribution of reinforcement in the solidified casting, particles must be engulfed in the primary matrix grains or dendrites during freezing. Engulfment of the reinforcement means that the particles are unlikely to be associated with brittle intermetallic phases and other particles in the interdendritic and inter-granular regions. If engulfment takes place it also suggests that reinforcement "wetting" has occurred and that the interfacial bonding between the particle and the solid matrix will be good [8]. Strong interfacial bonding is essential for effective load transfer from the matrix to the particle, it has a profound effect on the stiffness of the composite and delays the onset of particle–matrix decohesion.

For particle engulfment to occur independently of interface growth conditions, the thermodynamic driving force for pushing [9, 10] should be eliminated. It has been remarked that for a particle to have an affinity for the growing solid and hence engulfment to be preferred, the interfacial energy conditions must be similar to those for nucleation of solid on a particle substrate [11]. Since, via a direct thermodynamic approach, it is difficult to predict which types of particles will be engulfed, it seems sensible to assume that particles known to act as grain refiners for liquid metals are unlikely to be pushed during freezing.

1.4. Experimental aims

The aim of this experimental work is to produce Al-matrix composites with uniform, un-clustered spatial distributions of the reinforcing phase and to assess their mechanical properties. It was hoped that this could be achieved by facilitating spontaneous incorporation of reinforcements that encourage grain refinement.

2. Experimental procedure

2.1. Wetting agent

In order to facilitate the incorporation of reinforcing particles into molten aluminum, a novel composite fabrication process was adopted [6, 12]. This technique uses a K-Al-F-based flux, which is a mixture of $KAlF_4$ and K_3AlF_6 corresponding to the 45 mol % AlF_3 eutectic composition in the KF-AlF_3 system.

2.2. Reinforcement and matrix combinations

Two different types of reinforcement, TiB_2 and TiC particles, were investigated. It has been established [13–15] that these particles grain refine Al and Al alloys and this is why they were chosen. The mean size, density and source of these particulates are given in Table I. Reinforcements were added to commercial purity aluminum (approximate composition 99.7 wt %Al, 0.15 wt %Si, 0.15 wt %Fe). The different combinations of reinforcement and matrix investigated are outlined in Table II.

TABLE I Particle sizes and sources for the reinforcements investigated

Particle	Source	Density ($kg\,m^{-3}$)	Average particle size (and approx. size range)
TiB_2	Advanced Refractories Technology	4500	7 μm (1–30 μm)
TiC	London and Scandinavian Metallurgical Co. Ltd	4900	5 μm (3–9 μm)

TABLE II Combinations of reinforcement type and volume fraction examined

Base Alloy	Reinforcement (vol %)	Reinforcement type
99.7% Al	0, 5, 10	TiB_2
99.7% Al	0, 5, 10	TiC

2.3. Fabrication procedure

Metal charges were heated in a tube furnace to a temperature between 750 °C and 800 °C in clay-bonded SiC crucibles. The reinforcement powders were briefly tumble blended with an appropriate quantity of flux, to ensure an intimate dispersion, prior to the powder mixture being sprinkled onto the surface of the melt. The charge was then left unstirred for between 2–5 min allowing the flux to form a liquid and the reinforcement to be transferred into the melt. The liquid flux, which floats on the melt surface, was then removed using a ladle. After cleaning, the charge was briefly stirred to homogenize the dispersion of the particles and then cast. For these trials, composite melts of approximately 1 kg were made. The entire fabrication process, from initial addition to the point of casting, took no longer than 15 min.

2.4. Casting of the composite

The fluidity of composites containing 10 vol % of particle additions and below was not discernibly different from that of the un-reinforced base metal and, as a result, melts could be cast into a variety of molds. For the purpose of this investigation, charges were cast into a wedge-shaped mold (75 mm wide, 130 mm high, of angle 16°) to ascertain the effect of different cooling rates on the matrix microstructure and the distribution of reinforcement in the solid.

2.5. Metallographic examination

Macroscopic sections were taken through the thickness of the wedge, at the mid-point, along the full length of the casting, polished to reveal the macroscopic particle distribution and etched in Tucker's reagent to observe the overall effect of particle addition on the grain structure.

Samples for microscopic analysis were taken from the mid-plane between 15 and 40 mm from the tip of the wedge (where the cooling rate is greatest) and between 65 and 85 mm from the tip. Metallographic samples were polished and viewed in an optical microscope to reveal the particle distribution and anodized in Barker's Reagent and viewed in a polarizing light microscope to reveal the grain structure. Grain sizes at the 20 and 70 mm positions were measured using a line intercept method from five random straight lines drawn on micrographs of the specimens. Previous experimentation [6] has shown that the cooling rate of the melt is approximately 60 $K s^{-1}$ at the tip of the wedge, dropping to 8, 1 and 0.5 $K s^{-1}$ at positions 20, 70 and 85 mm from the tip, respectively. This variation in the cooling behavior with position was assumed to be characteristic of the mold and, therefore, reproducible from casting to casting.

2.6. Mechanical testing

Mechanical testing was performed on both 10 vol %-reinforced composite and unreinforced samples taken horizontally, along the width of wedge castings, between roughly 40 and 60 mm from the tip. The modulus was measured and averaged over three loading cycles using a twin strain gauge method outlined by Clyne and Withers [16]. The 0.2% proof stress, ultimate tensile stress and ductility were also measured. The results in each case represent an average of a minimum of six test samples. Densities of the base material and the composites were measured using an Archimedian technique in order to determine the particle volume fraction and from that, the particle yield.

3. Results

3.1. Particle incorporation

When the flux was added to the surface of the molten Al a liquid was produced, which rapidly enveloped the reinforcement, enabling transfer of the particles to the melt. It was impossible to incorporate TiB_2 and TiC particles into Al in this way without the use of a flux, even using a moderate level of stirring. Whilst for both types of particles the density measurements in Table III indicate that close to 100% of the particles added were successfully incorporated, the TiC particles had a slightly higher yield and separated more thoroughly and rapidly from the flux to produce cleaner composites. The entrainment of black flux inclusions into the castings is apparent in Figs 1 and 2, in particular for TiB_2-reinforced composites. Flux inclusions and

TABLE III Composite densities (in kg m^{-3}), volume fractions and yields

	Density for 0 vol % addition	Density after 10 vol % addition	Calculated volume fraction	Approximate yield (%)
Al-TiB_2	2673	2843	0.093	93
Al-TiC	2673	2889	0.097	97

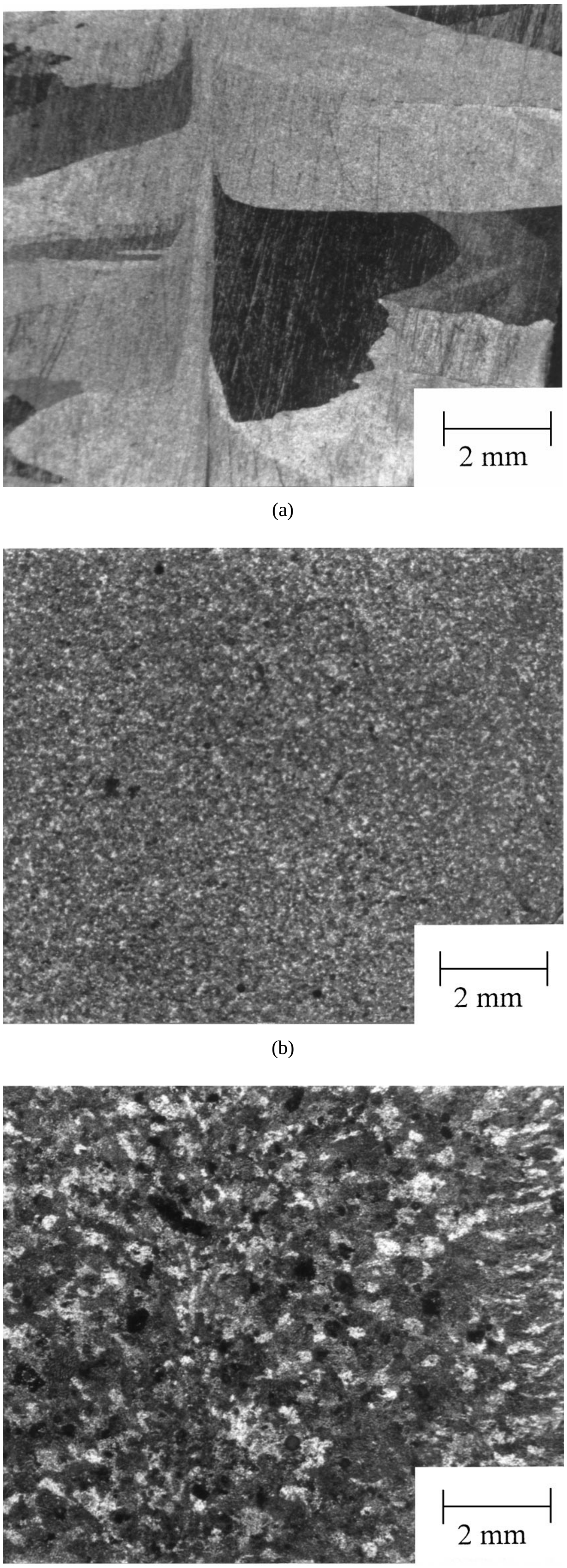
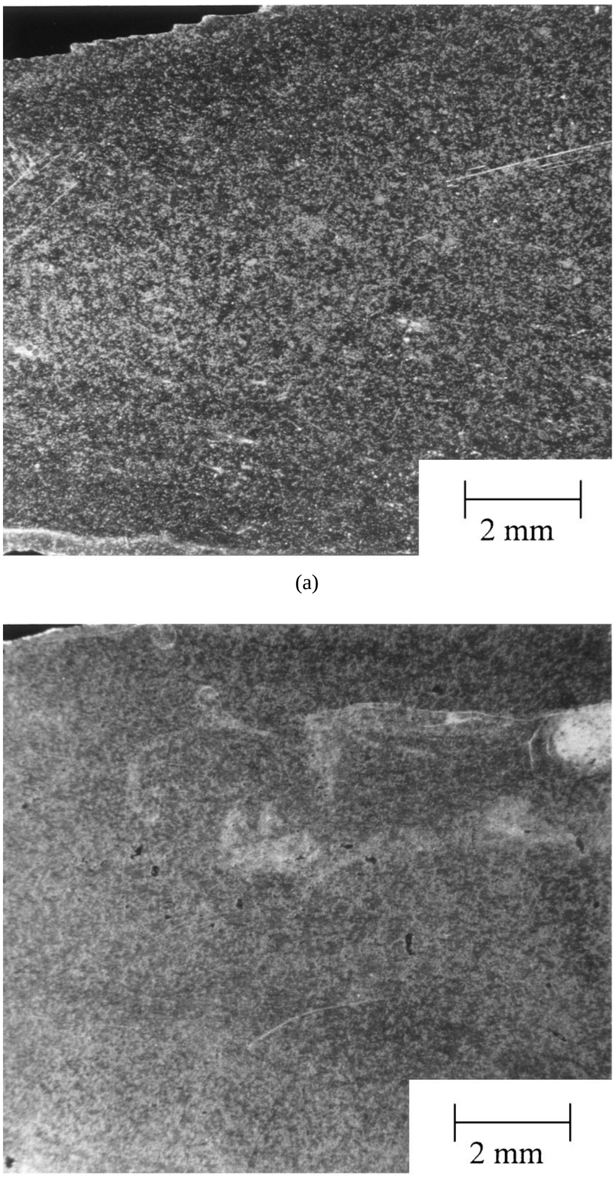


Figure 1 Macrographs showing the grain structure for (a) un-reinforced Al, (b) Al-5 vol % TiC, and (c) Al-5 vol % TiB_2 castings.



(a)

(b)

Figure 2 Macrographs showing the particle distribution in (a) 5 vol % TiC, and (b) 5 vol % TiB_2-reinforced composites.

TABLE IV Grain sizes (in μm) before and after particle additions

	99.7% Al	5 TiB_2	10 TiB_2	5 TiC	10 TiC
20 mm from tip	1500 ±250†	365 ±20	260 ±30	75 ±14	62 ±10
70 mm from tip	>4000†	560 ±80	475 ±65	112 ±20	92 ±18

†denotes highly elongated structures.

porosity will lower the apparent density of the composites thus making the yields presented in Table III lower bounds and the actual values closer to 100%.

3.2. Grain structure in the wedge castings

3.2.1. *Un-reinforced Al*

The macrostructure of the un-reinforced casting varies strongly with position, increasing in size with distance from the wedge tip. Grain size measurements presented in Table IV indicate that the grain size is very large and more than doubles in size between the front and the back of the wedge. Throughout the casting the grain structure is highly elongated. The macrostructure around the 40 mm position is shown in Fig. 1a.

3.2.2. *TiC-reinforced Al*

The presence of TiC particles results, as expected, in considerable grain refinement. Fig. 1b presents the macrostructure observed in a 5 vol % TiC composite, at a position 40 mm from the wedge tip, and shows that the grain structure is highly refined. This refinement occurs over the full length of the casting. The grain size measurements presented in Table IV indicate that there is only a small variation in the grain size with position in the wedge casting. This table also illustrates that as the volume fraction of particles added increases, the composite grain size decreases. The grain size is only slightly smaller after 10 vol % additions, suggesting that at higher particle fractions a smaller proportion of the particles act as grain nucleation sites.

3.2.3. *TiB_2-reinforced Al*

The addition of TiB_2 also causes grain refinement, as shown in Fig. 1c. The grain size reduction is not as great as that after TiC addition and data in Table IV indicate that cooling rate influences the grain size more significantly. This table also shows that higher particle fractions produce increased refinement but again, as is the case for TiC, the effect is not in proportion to that caused by the initial addition. The presence of flux inclusions is particularly clear in these macrographs and it is apparent that there is more included flux in the TiB_2-reinforced system.

3.3. Particle distribution

There was no significant macroscopic (long-range) TiC or TiB_2 particle redistribution associated with particle pushing in from the side-walls of the mold in any of the sectioned wedges. Macrographs in Fig. 2 show this for 5 vol %-reinforced composites. Particle distributions in 10 vol %-reinforced composites are presented at higher magnification in Fig. 3 and it is clear from these micrographs that the TiC particles are more evenly spaced and form far fewer large clusters.

3.4. Relationship between particle distribution and grain structure

3.4.1. *TiC-reinforced Al*

As shown in Fig. 4, the distribution of the TiC reinforcing phase is relatively uniform and un-clustered, with the particles being located mainly in, but also around, the metal grains. In qualitative terms, the homogeneity of the particle distribution does not vary significantly as the cooling conditions change, since the microstructure

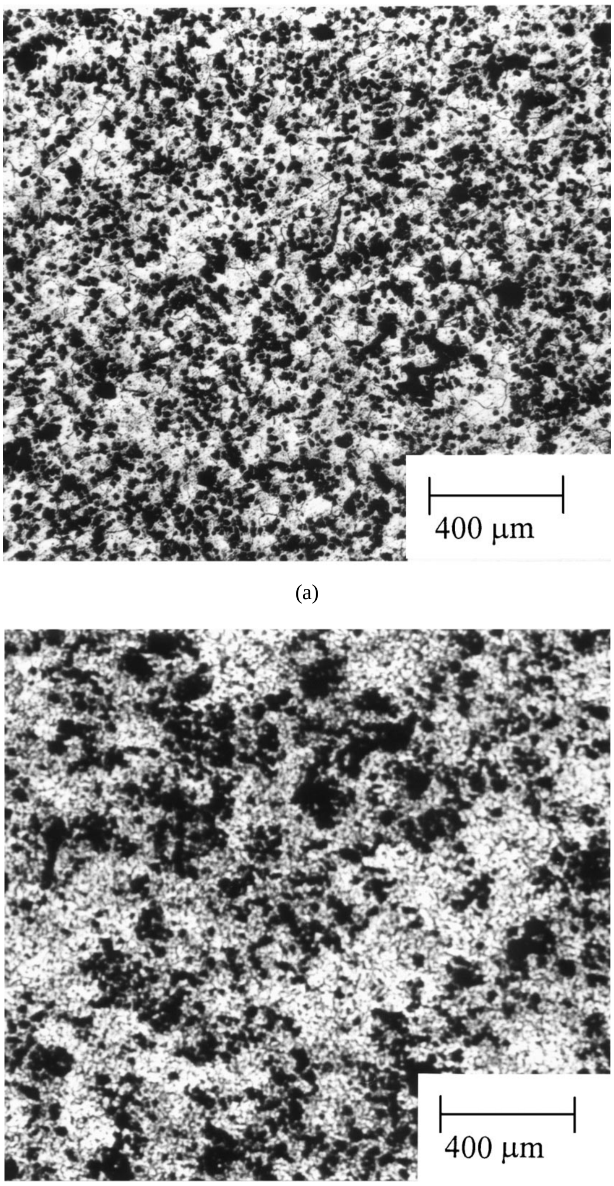


Figure 3 Optical micrographs showing the particle distribution in (a) 10 vol % TiC, and (b) 10 vol % TiB_2-reinforced composites.

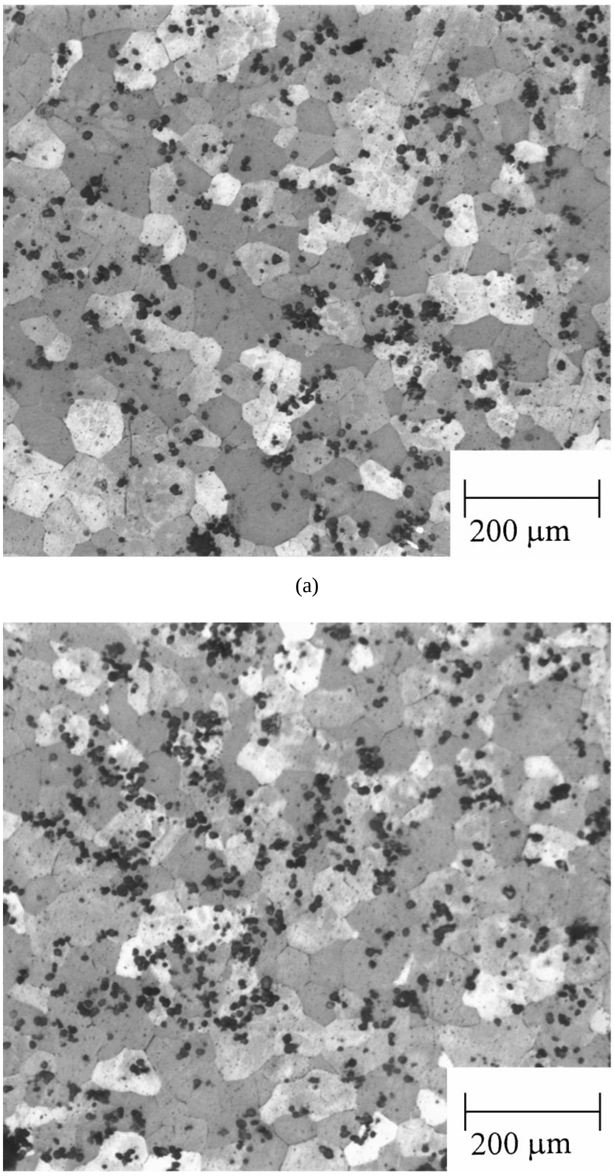


Figure 4 Micrographs showing the grain structure in a 5 vol % TiC-reinforced composite cooled under (a) fast, and (b) slow conditions.

is hardly affected by the cooling rate. The only detectable feature is that at higher volume fraction additions, as shown in Fig. 6a, a smaller percentage of the particles stimulate grain refinement and hence a smaller proportion of them are located at the center of the matrix grains. Despite this, there are very few large particle clusters or particle-free regions.

3.4.2. TiB_2-reinforced Al

Fig. 5 shows that the majority of the TiB_2 particles and clusters have been engulfed within the large grains to produce a reasonably uniform, intra-granular distribution of reinforcement. There is a larger spread in the particle size range for the TiB_2 particles, but both the largest and the smallest particles appear to be engulfed. Once again changes in the cooling rate seem to have little effect on the spatial distribution of the reinforcement. As the particle fraction increases, however, the tendency for agglomeration increases, as shown in Fig. 6b, and, although the TiB_2 particles are engulfed, they are not as evenly distributed as the TiC particles.

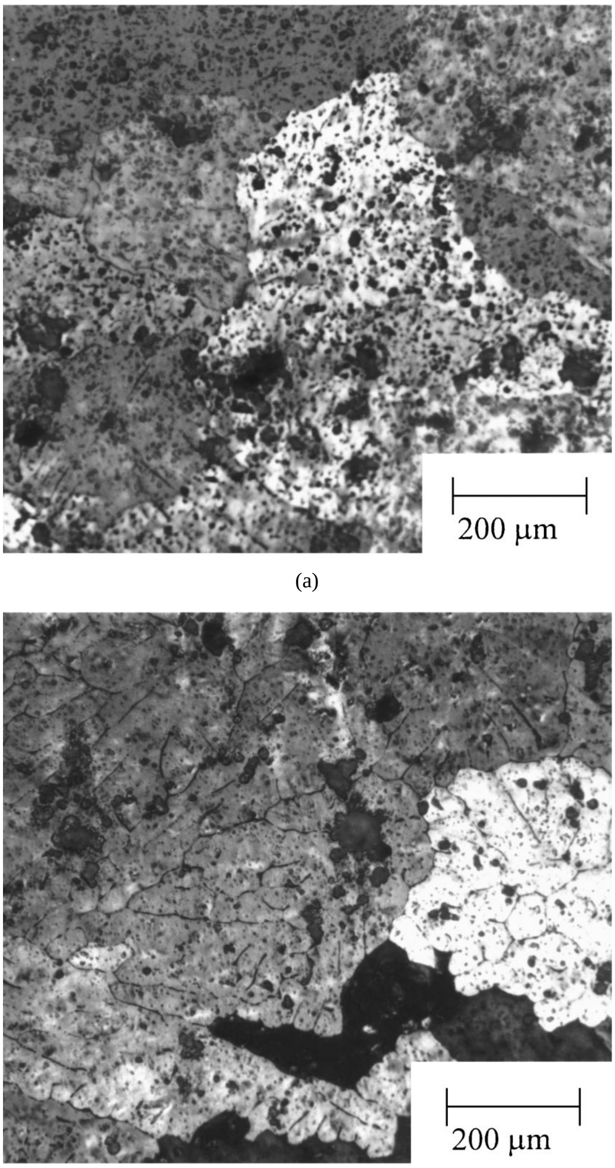
3.5. Mechanical testing

Table V presents mechanical property data for samples from the wedge castings. As expected, the addition of reinforcement produces increases in strength and stiffness over the base alloy at the expense of ductility. The addition of TiC particles produces composite mechanical properties superior to those resulting from TiB_2 particle addition. Both composites, however, retain a high level of ductility, particularly for cast material.

4. Discussion

4.1. Particle incorporation

It is thought that the K-Al-F, cryolite-type, flux facilitates particle incorporation by dissolving the oxide layer on the surface of the melt, as it is likely to have a high solubility for Al_2O_3 [17, 18]. The removal of the



(a)

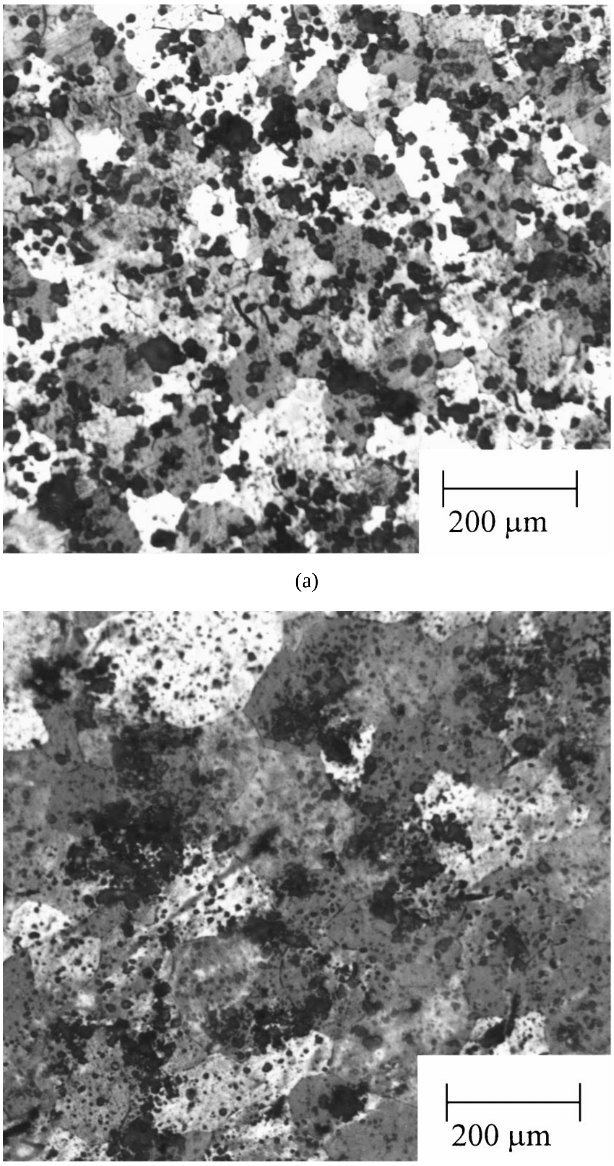
(b)

Figure 5 Micrographs showing the grain structure in a 5 vol % TiB_2 reinforced composite cooled under (a) fast, and (b) slow conditions.

TABLE V Mechanical properties for the base alloy and composites

	Modulus (GPa)	0.2% P. S. (MPa)	UTS (MPa)	Ductility (%)
99.7% Al	69 ±1.0	33 ±2	66 ±3	37 ±2
99.7% Al 10-TiC	89 ±1.0	63 ±3	105 ±6	25 ±3
99.7% Al 10-TiB_2	85 ±1.0	51 ±4	80 ±5	18 ±2

melt oxide layer not only eliminates the mechanical obstacle to particle entry, but will improve particle–melt wetting. The contact angle for Al on TiB_2 is roughly 150° at 690 °C and 98° at 900 °C when a surface oxide film is present [19]. Similarly, the wetting angle for TiC on an oxidized melt surface is 118° at 700 °C [20]. This suggests that spontaneous entry is impossible without fluxing agents. The contact angle for TiB_2 is reported to reduce to approximately 67° at 800 °C [19] and that for TiC to reduce to 10° at 990 °C, after the Al oxide film has either been eliminated or disrupted [20], implying that spontaneous entry is possible with the oxide barrier removed. The spontaneous transfer of TiB_2 and TiC into aluminum melts when a flux is used confirms that



(a)

(b)

Figure 6 Optical micrographs for (a) 10 vol % TiC, and (b) 10 vol % TiB_2-reinforced composites.

with the melt oxide layer removed, liquid aluminum will wet both these ceramics.

In this study, a slight difference between the transfer rates for TiB_2 and TiC particles was observed under otherwise identical conditions, TiC particles being slightly more readily incorporated. This can be explained by improved wetting of TiC by Al, since it has been remarked that compounds with less negative heats of formation wet better [20] (the heat of formation for TiC at 298 K is $-184\ \mathrm{kJ\,mol^{-1}}$ and that for TiB_2 is $-324\ \mathrm{kJ\,mol^{-1}}$ [21]). The enhanced affinity TiC particles have for the melt may also explain their improved separation from the flux and the cleaner composites that result from this. Since the particles are of a similar size and density, it is unlikely that these factors contribute to significant variations in the incorporation behavior.

4.2. Particle agglomeration

The absence of extensive TiC agglomeration suggests that in this system the thermodynamics for adhesion are unfavorable. Small-scale clustering of TiB_2 particles was observed and this supports the postulate that molten Al does not wet TiB_2 particles as well as it wets TiC particles. More extensive agglomeration at higher particle fractions is due to the increased likelihood of inter-particle collisions with greater numbers of particles present.

4.3. Composite microstructure and particle distribution

In the Al-TiC system there is thought to be sufficient lattice matching for the direct nucleation of solid Al to occur on the particle surfaces [22, 23] and grain refinement and particle engulfment are expected. At higher particle fractions, the nucleation efficiency, the proportion of active nucleation sites, seems to decrease. This is not unexpected as it is much easier for atoms to attach to one of the many growing crystals than for more nucleation sites to become active [24]. It is also believed that when many grain nuclei are present and growing, recalescence suppresses further grain nucleation [25]. The net result is that for larger volume fraction additions, not all the TiC particles are engulfed to reside in the center of the Al grains and a greater proportion are located at the grain boundaries.

There is good atomic spacing matching between the HCP TiB_2 structure and the FCC Al structure if the (0 0 0 1) and (1 1 1) planes, respectively, are considered. This would suggest that grain refinement is possible. There is, however, a large difference in the extent of grain refinement resulting from similar additions of the two different particle types. In the Al-TiB_2 system there are fewer sets of closely matching planes than in an isomorphous melt and particle system (such as Al and TiC) and this could explain why the nucleation efficiency is much lower and grain refinement is not so prominent. Whilst relatively few TiB_2 particles act as grain nucleation sites, it is clear that they have an affinity for the solid phase and that engulfment is favored.

For both particle types, it appears that particle pushing has largely been suppressed and that engulfment of the particles into the growing solid has been encouraged. In the case of the TiC particles, the resultant grain size is equi-axed and very small, and for this reason it is very unlikely that long-range particle pushing can take place to generate significant distributional heterogeneities. Since grain refinement will occur under even slower cooling conditions than those encountered in this study, it is unlikely that long-range particle pushing will ever occur in this system.

In the TiB_2-reinforced composites, the grain size is large compared to the inter-particle spacing. If pushing were favorable in this system, it could occur over large enough distances to create significant heterogeneities. Engulfment was, however, observed over the range of cooling rates experienced in this work. At higher particle fractions, where we would expect pushing to become more difficult, significant particle clusters were observed. These clusters were located within the metal grains suggesting that they were engulfed and that they were created due to agglomeration of particles in the melt before freezing took place. It is possible that under slower cooling conditions, and for smaller TiB_2 particles, both short and long-range particle pushing could occur.

The interfacial energy between the particle and the solid metal seems to dictate the condition for engulfment. Grain refinement, however, does not appear to be a prerequisite for particle engulfment but, it should be noted that to achieve good spatial distributions of the reinforcing phase regardless of the cooling conditions, grain refinement is desirable.

4.4. Mechanical properties

Modulus increases of $2\,\mathrm{GPa}\,(\mathrm{vol}\,\%)^{-1}$ of TiC added and $1.6\,\mathrm{GPa}\,(\mathrm{vol}\,\%)^{-1}$ of TiB_2 added, compare favorably with those for cast Al_2O_3 ($1.2\,\mathrm{GPa}\,(\mathrm{vol}\,\%)^{-1}$) and SiC ($1.3\,\mathrm{GPa}\,(\mathrm{vol}\,\%)^{-1}$) particulate-reinforced systems [26, 27]. These reinforcements have similar, if not higher, stiffnesses, 430 and 450 GPa respectively, than TiC (230–400 GPa) and TiB_2 (350–570 GPa) [28], and hence we would expect them to induce greater stiffening. The fact that TiC and TiB_2 additions produce larger stiffness improvements suggests that interfacial bonding is stronger in these systems. It is thought that this improved interfacial bonding is achieved through wetting of the reinforcement by the liquid, and hence spontaneous incorporation, and particle engulfment into the solid phase, due to the low particle-solid interfacial energy. These two events are seldom observed in the Al-SiC and Al-Al_2O_3 systems.

Of the two composites produced in this study, those containing TiC reinforcement have better mechanical properties than those with TiB_2 additions. Improvements in 0.2% proof stress for TiC additions could be attributed to the reduced grain size. According to Sahoo and Koczak [29] a reduction in the grain size from 475 μm to 92 μm corresponds to a yield strength increase of 11 MPa, an improvement which is consistent with the 12 MPa increase observed in this work.

A significant difference in the modulus was observed (4 GPa) and this can not be attributed to small (maximum of 0.004) variations in the volume fraction of reinforcement present in the two composites. The difference is more likely to be due to improved interfacial bonding in the Al-TiC system. This is probably brought about by better wetting by the liquid phase, which will eliminate the presence of adsorbed gas and flux at the particle-liquid interface, and the increased tendency for nucleation of solid on the particle surfaces. Higher ductility was also observed in the Al-TiC system and this is probably due to a combination of the less clustered particle distribution and improved interfacial bonding.

5. Conclusions

TiB_2 and TiC particles have been spontaneously incorporated into commercial purity (99.7%) aluminum melts using fluxing agents which remove the melt surface oxide and promote contact between clean, wetting surfaces.

TiC particle additions significantly refine the grain structure whilst TiB_2 particles only stimulate mild refinement.

For both TiC and TiB_2 particle additions, particle pushing is largely suppressed, causing the majority of the particles to become engulfed into the growing solid. It is expected that particle engulfment is facilitated due to close crystal structure matching and, therefore, the low particle-solid interfacial energies in the Al-TiC and Al-TiB_2 systems.

There appears to be little driving force for agglomeration of the TiC particles but small TiB_2 clusters were observed, suggesting that TiB_2 particles are wet less well by molten Al.

Modulus increases resulting from TiC and TiB_2 particle additions to molten Al are greater than those for Al_2O_3 and SiC additions. This can be explained by stronger interfacial bonding in the TiC and TiB_2 systems due to wetting of the reinforcement by the liquid and particle engulfment into the solid phase.

TiC-reinforced composites have higher stiffnesses and ductilities than those to which TiB_2 particles have been added. This is thought to be due to improved interfacial bonding in the Al-TiC system caused principally by the increased tendency for nucleation of solid on the particle surfaces.

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