

Solidification Observations of Dendritic Cast Al Alloys Reinforced with TiC Particles

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TiC particulate composites in the range of 3 to 5 vol.% were prepared by diluting Al/20 vol.% TiC composite in Al7Si, Al20Cu, and Al20CuNi alloy matrices. TiC particle distribution consists of isolated particles located both at the primary α -dendrites and the areas of the lastly solidified liquid and particle clusters located at the grain boundaries. Particle-solidification front interactions were responsible for the final particle location and the mechanisms of such behavior were analyzed. The solidified microstructure consists of primary, eutectic, and other intermetallic phases, with the latter being associated with the presence of TiC particles.

Keywords Al alloy cast structures, aluminum matrix composites, particle-melt interactions, particle-solidifying front interactions, TiC particulates, wetting

1. Introduction

Particulate-reinforced aluminum matrix composites (PRAMCs) have been at the forefront of the technological and scientific interest for more than four decades due to their potential as attractive candidate materials for many applications such as in the aerospace, automotive, and structural application sectors. Property improvements such as strength and stiffness, density, abrasion, and wear resistance seem to justify this continuous development frame of such materials (Ref 1-5).

Many different processing routes have been exploited for the production of PRAMCs, based on either liquid or solid matrix state. Among these processes, the stir casting process has always been the subject of extensive research activity, due to its low cost and ease-to-handle considerations. A great variety of phenomena are governing the reinforcement-matrix possible interactions, and extensive reviews on such aspects can be found in the works of Rohatgi et al. (Ref 2), Mortensen and Jin (Ref 3), and Asthana and Tewari (Ref 5). It is, however, important to address the main aspects that influence the final microstructure of a cast composite, since it is the manufacturing process adopted and the main subject of the present effort. The two fundamental phenomena involved in cast metal matrix composites are the particle-liquid metal interactions and the particle-solidification front interactions.

The particle-melt interactions are responsible for both the introduction of the particulate reinforcement within the melt and the primary particle distribution in the liquid metal. Good

compatibility between particles and molten matrix results in successful reinforcement insertion and uniform distribution. The concept of good compatibility is assessed by means of wettability, i.e., the ability of a liquid to spread over the surface of a ceramic substrate to form a low ($<90^\circ$) contact angle, in the classic so-called sessile drop experiment. In terms of particle transfer within the melt, this low contact angle condition is not, however, the absolute remedy for reinforcement insertion. Total spreading (0° contact angle) is required instead, if spontaneous entry is demanded. In practice, the favorable wetting characteristics of less than 90° contact angle are supported by the application of mechanical stirring that provide the necessary shear forces for total particle insertion. Oxide or gas phases can poison the involved surfaces. Especially in the case of Al, the presence of surface oxide phase can alter the reinforcement-melt interactions and may obstruct them from expressing their real wetting characteristics. On the other hand, reinforcement particle insertion can be more favorable in the absence of surface oxide phase (Ref 6, 7).

The particle-solidification front interactions influence the final particle distribution and location. The particles can be pushed by the solidification front, engulfed by the solidification front, or act as solid phase nucleation sites. Many theories have been developed concerning the solidifying front-reinforcing particle interactions, which are based on thermodynamic and kinetic considerations. Zubko et al. (Ref 8) proposed the thermal conductivity criterion, according to which engulfment will occur when the thermal conductivity of the liquid is higher than that of the reinforcing particles. This theory was refined by Surrapa and Rohatgi (Ref 9). Many other theories have been based on the thermal conductivities and the thermal fields developed during the solidification of cast composites such as in the works of Rohatgi et al. (Ref 10), Hadji (Ref 11), and Garvin and Udaykumar (Ref 12), while the importance of other factors such as interfacial characteristics and crystallographic compatibility are addressed in the works of Kaptay (Ref 13, 14), Ohta and Suito (Ref 15, 16), and Schaffer et al. (Ref 17). An analytical model for the interaction between an insoluble particle and an advancing solid/liquid interface has been presented by Shanguan et al. (Ref 18); in this model, they

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addressed the concept of the critical velocity of the interface, V_{cr} , below which the particle will be pushed and above which the particle will be engulfed. They also showed the dependence of V_{cr} on surface energy changes and particle size.

Many other models have been based on the concept of solidification front critical velocity (Ref 19-36) such as in the works of Stefanescu et al. (Ref 25), Han and Hunt (Ref 26), Juretzko et al. (Ref 27), Shibata et al. (Ref 28), Kimura et al. (Ref 29), Catalina et al. (Ref 30), Mukherjee and Stefanescu (Ref 31), Mukherjee et al. (Ref 32), Youssef et al. (Ref 33), Garvin et al. (Ref 34, 35), and Stefanescu (Ref 36). Stefanescu and Dhindaw (Ref 37) summarized the parameters affecting the critical velocity for the different cases of solidification conditions (unidirectional-multidirectional) and the different morphologies of the solidification front (planar, cellular, and dendritic). They concluded that V_{cr} can be described qualitatively as

$$V_{cr} = f(\Delta\sigma, 1/\eta, 1/r, K_l/K_p, G),$$

where $\Delta\sigma$ is the change in the interfacial energies, η is the viscosity of the melt, r is the particle radius, K_l and K_p are the thermal conductivities of the liquid and the particle, respectively, and G is the temperature gradient ahead of the solidification front. For summarized approaches on the phenomena concerning the interaction of insoluble particles with the solidification front in cast metal matrix composites, the reader is strongly recommended to refer to the review works of Asthana and Tewari (Ref 38) and Asthana (Ref 39).

Although a variety of Al alloys have been investigated as potential matrix systems (Al, Al-Si, Al-Mg, Al-Mg-Si, Al-Cu, etc.), the research works have mainly been concentrated on SiC and Al_2O_3 particulate reinforcements (Ref 1-5). However, several experimental efforts have been focused on the utilization of TiC particles as potential reinforcements of Al alloys. Characteristic examples that can be considered are as follows: the works of Contreras et al. (Ref 40) and Albiter et al. (Ref 41) who produced Al-Mg/TiC and Al-2024/TiC composites, respectively, by melting infiltration techniques; the work of Selcuk and Kennedy (Ref 42) who prepared an Al/TiC master composite of high TiC content (over 50 vol.%) by reaction synthesis of elemental powders; the works of Shyu and Ho (Ref 43), Premkumar and Chu (Ref 44), Kharti and Koczak (Ref 45), and Sahoo and Koczak (Ref 46) who produced Al/TiC composites through the reaction of carbonaceous gas with Ti dissolved in an Al melt; and the work of Karantzalis et al. (Ref 7) who produced Al/TiC composites by a stir casting process.

TiC particles were chosen as the reinforcing phase because of its properties and metallic characteristics (Ref 47, 48), which is an important factor for sufficient wetting when in contact with aluminum melts without the necessity of interfacial chemical reactivity. Such behavior can lead to great affinity for molten aluminum, reduced tendency for particle agglomeration, good particle distribution in the melt, and development of strong interfaces, free of reaction products.

Three different alloy matrices were facilitated in this work: (a) an Al-7Si hypoeutectic alloy with 0.25 to 0.5 Mg, Sr modified, (b) an Al-20Cu hypoeutectic alloy with the main impurity being Fe, and (c) an Al-20Cu-Ni alloy with 2 to 3% Ni. All compositions are in wt.%. The scope of the present effort was to investigate the behavior of TiC particles during solidification. The first two matrices experience same morphological features during casting, whereas the addition of Ni in the

third one provides the opportunity to examine the potential of TiC particles to act or not act as potential nucleation sites for intermetallic phases.

2. Experimental Procedure

The examined composites were prepared by diluting a quantity of a master composite material in the main matrix alloys presented previously (Al-7Si hypoeutectic alloy Sr modified, Al-20Cu hypoeutectic alloy, Al-20Cu-Ni alloy). The master composite was Al-20 vol.% TiC prepared by a flux-assisted stir casting process. The preparation method involved the addition of the appropriate mixture of KBF_4 flux along with TiC powders (-325 mesh, <44 μ m) onto the surface of the Al melt. The halide salt reacts with Al and forms a layer of slag that dissolves the oxide film from the melt surface. Subsequently, TiC particles and Al melt express their net wetting characteristics, allowing the spontaneous insertion of the reinforcement within the molten metal. Yet, the exact details and the scientific background of the technique are a matter of a future publication. The amount of the master composite added was such that to produce a final 3 to 5 vol.% TiC composite material. The matrix material was heated up to 800 °C in a resistance furnace. The master composite was then added and allowed for few minutes (not more than 5 min) to dissolve and release the TiC particles. The melt was then gently and softly stirred to prevent particle settling due to sedimentation. Finally, the slurry was poured into steel cylindrical molds. Specimens were cut, mounted, and prepared for inspection.

Standard metallographic procedures were applied (grinding by 80, 200, 500, 800, and 1000 SiC grit papers followed by polishing by 6, 3, and 1 μ m diamond suspensions). Etching was performed with 10% HNO_3 aqueous solution. Metallographic inspection was conducted with a LEICA 4000 optical microscope. Particle content measurements were conducted with the aid of 'Image J', the image analysis software. The technique is based on the calculation of the different area portions of the involved phases, which are colored differently, through the use of the appropriate optical microscope images. SEM inspection was carried out using the JEOL 5600 system equipped with an Oxford Instruments EDS analysis system.

3. Results and Discussion

3.1 Particle Incorporation

The incorporation of ceramic particles into the molten alloys examined in this study was approached by diluting appropriate amount of a master composite material into certain amount of melt load. Such an approach provides some advantages as follows: (a) The ceramic particles have already been incorporated in an aluminum matrix and hence no significant contact between them and the melt surface was experienced. In such a way, poisoning of the particles by oxide phases is eliminated if not avoided. (b) The characteristics of the production method of the master composites are such that they ensure a good TiC-Al wetting behavior. The master composite has been produced by

a casting method where fluxing agent/salt, KBF_4 salt, is added onto the melt surface mixed with the TiC particles. The role of this ingredient is extremely important since it reacts with molten Al, forming a slag layer that dissolves the surface oxide, allowing reinforcing particles and molten alloy to express their net wetting characteristics and making the particle entry more favorable. The real benefit of such salt treatment is the subsequent formation of clean, unpoisoned ceramic-melt interfaces. Therefore, for the adopted production route, no rigorous stirring or high-temperature processing is required.

3.2 Particle Distribution

Figure 1(a) to (c) shows a panoramic view of the microstructure of the solidified A356-, Al20Cu-, Al20CuNi-TiC composites, respectively. Image analysis has revealed that the reinforcement is about 3 to 5 vol.% in all cases. The ceramic phase is relatively uniformly distributed both as isolated particles and as medium-sized clusters. Figure 2(a) to (c) presents the microstructure of the same alloys under higher magnification and after etching. It can be observed that primary α -dendrites, in all cases, have similar grain size of the order of 80 to 100 μm . Figure 3(a) to (c) provides more details on ceramic particle location. Two separate cases can be distinguished: isolated particles that are located both at the interior of the primary dendrites and the grain boundaries and particle clusters that are mainly located at the grain boundaries and the areas of the lastly solidified liquid. In this case, the particles are associated with the presence of eutectic phases and, especially in the case of Al20CuNi alloy (Fig. 3c), with other intermetallic phases.

The final distribution and location of the ceramic particles is a function of two important phenomena: the interaction between particles and molten alloy and the interaction between ceramic particles and solidifying alloy. These two interactions are also responsible for the overall microstructure and the interfacial characteristics of the final composite materials and will be addressed in detail in the following paragraphs.

3.2.1 Particle-Melt Interactions. The uniform dispersion of the ceramic phase, both as isolated and clustered particles, in the molten matrix is a crucial factor for the resulting microstructure and is directly linked with the wetting characteristics of the involved phases. Good wetting is primarily responsible for the introduction of the ceramic phase into the molten alloy. TiC is a compound of strong metallic nature owing to the presence of Ti. Such a metallic characteristic is reported to improve the wetting behavior of TiC when in contact with metallic melts (Ref 47, 48). The Al-TiC wetting behavior is somehow more complicated. In general, the system is characterized by good wettability, low contact angles, and good interfacial bonding (Ref 49). However, such behavior is achieved only when the surface oxide layer covering the Al melt is removed or dissociated. This removal or dissociation occurs only at high temperatures ($>900\text{ }^\circ\text{C}$) (Ref 31). At such high temperatures, the system becomes intensively reactive; the dissociation of TiC and the formation of Al_4C_3 and/or Al_3Ti compounds take place at or close to the interfacial area (Ref 47, 50). In general, such reactivity improves wettability (Ref 47, 48); however, the formation of new phases can alter dramatically the nature and the response of the reinforcement-matrix interface. Such formation has not been observed in this work. This is probably attributed to the short processing time.

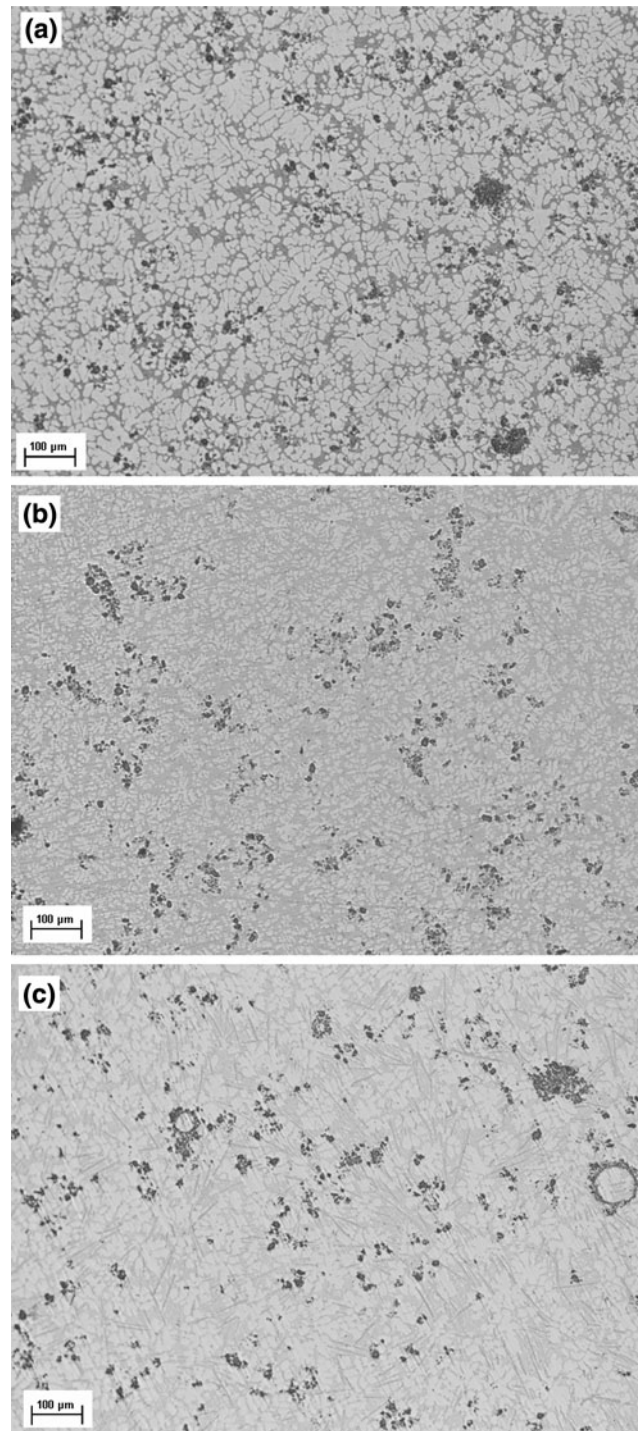


Fig. 1 Optical micrographs presenting a panoramic view of the produced composite materials. Reinforcing content is estimated to lay between 3 and 5 vol.% in all cases. Ceramic particles are distributed uniformly as both isolated and medium-sized particles. (a) Al7Si, (b) Al20Cu, and (c) Al20CuNi matrices

The use of fluxes in the production of the master composite ensures minimization of the oxide phases (Ref 6, 7) and consequent improvement of the wetting behavior. This beneficial action is attributed to the ability of the formed slag to dissolve the oxide phases present on the surface of the

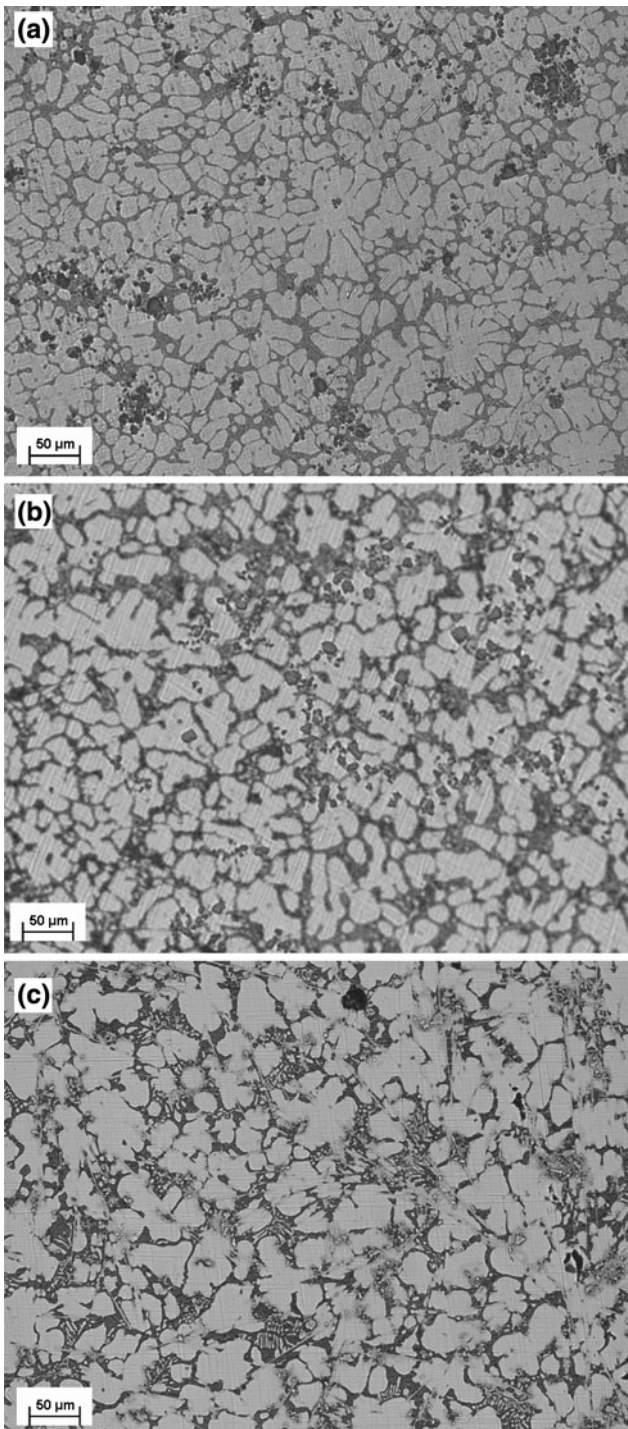


Fig. 2 Optical micrographs showing the grain size of the various composites being 80 to 100 μm in all cases. (a) Al7Si-, (b) Al20Cu-, and (c) Al20CuNi-based composites. Etching was performed with 10% HNO_3 aqueous solution

melt. Such a good wetting may lead to a good particle distribution within the molten alloy. The clusters observed could either be inherited by the precursor master composite or be a result of the solidifying front-particle interactions, as discussed in Section 3.2.2.

During conventional manufacturing of cast metal matrix composites, extensive mechanical stirring is used to provide the

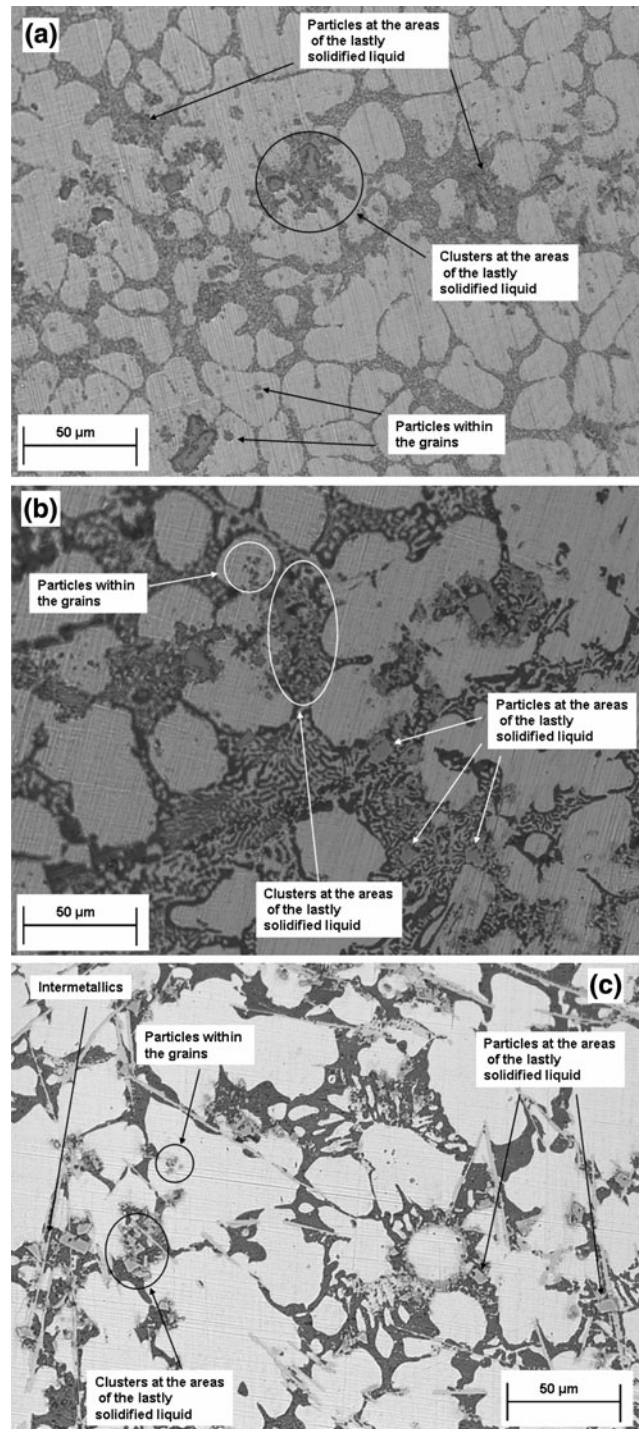


Fig. 3 Optical micrographs showing particle location. Isolated particles are located both with the primary dendrites and the grain boundaries. Clustered particles are mainly located at the areas of the lastly solidified liquid. (a) Al7Si-, (b) Al20Cu-, and (c) Al20CuNi-based composites

necessary shear forces for both reinforcing phase insertion and precursor material clusters dissociation. In this study, such a mechanical stirring was not adopted because the dilution process along with the method to produce the precursor master composite ensures the particle insertion without the necessity of external mechanical stirring.

3.2.2 Solidifying Front-Particle Interactions. Despite the particle-melt interactions, during the solidification process, the TiC particles are facing the solidifying front. This interaction also plays an important role in the particle distribution and the final microstructure.

As already mentioned in all the different cases of the produced composites, the final particle distribution basically comprises the location of both isolated and clustered particles at the lastly solidified liquid at the interdendritic areas as well as the location of isolated particles within the Al dendrites. Such particle distribution has been predicted and analyzed by Stefanescu and Dhindaw (Ref 37) when explaining the particle-solidification front interactions in the case of multidirectional, dendritic solidification. This particle location, according to this work, is a strong evidence that the critical solidification rate for particle engulfment has not been achieved. This critical solidification front velocity is a function of many factors. However, the inability to reach the critical value and hence to accomplish particle entrapment can be explained in thermal conductivity terms as described in the following paragraph.

The first phase to solidify is the α -Al phase that pushes without engulfing the majority of the TiC particles at the last to solidify interdendritic areas. In this case, the so-called “thermal conductivity” model that takes into consideration the thermal conductivities of the involved phases proposed by Zubko et al. (Ref 8) and refined by Surappa and Rohatgi (Ref 9) appears the most applicable. On the basis of the works of Poirier and McBride (Ref 51) and Brandt and Neuer (Ref 52), the thermal conductivities of the main Al-20Cu and Al-7Si alloys at their melting point are ~ 95 and ~ 80 W/m·K, respectively. The thermal conductivity of TiC is ~ 21 W/m·K. These thermal conductivity differences, in both cases, cause the liquid at the

vicinity of the hot ceramic particles to be at higher than the rest of the melt temperature during cooling. Hence, the solid phase prefers to start its nucleation away from the particle surface than at particles-free melt areas. This nucleation initiation causes the evolution of alloying elements at the molten alloy close to the vicinity of the primary Al grains. Such evolution can cause significant melt undercooling, resulting in a preferential growth of the melt through the liquid phase. This last observation inhibits even more any potential tendency of the TiC particles for heterogeneous nucleation. Consequently, the TiC particles are blocked within the last solidified liquid where eutectic and other intermetallic phases are formed. This TiC particle rejection at the interdendritic areas can also be a reason for cluster formation.

Another important observation that can be explained by the presence of TiC particles and the thermal conductivity differences is the refined structure of the solidified dendrites, by means of their equiaxed morphology. A nucleated Al grain starts to grow away from the TiC particles which, however, built up a front of high temperature liquid. In this way, a dendrite tip that approaches a ceramic particle realizes a disturbance on the thermal gradient ahead of the tip and its further growth eventually stops. The dendrite growth consequently has to continue toward other directions where supercooled liquid is available. This results in the splitting of the dendrite arm in more branches, leading to a finer and more equiaxed morphology of the final dendrites. The mechanism is presented schematically in Fig. 4. Such a mechanism has also been reported in the work of Zhou and Xu (Ref 53). Figure 5 shows two steps of TiC particle entrapment under SEM (back-scattered mode) examination.

It was presented previously that particles are either entrapped within the grains of primary α -Al or pushed at the

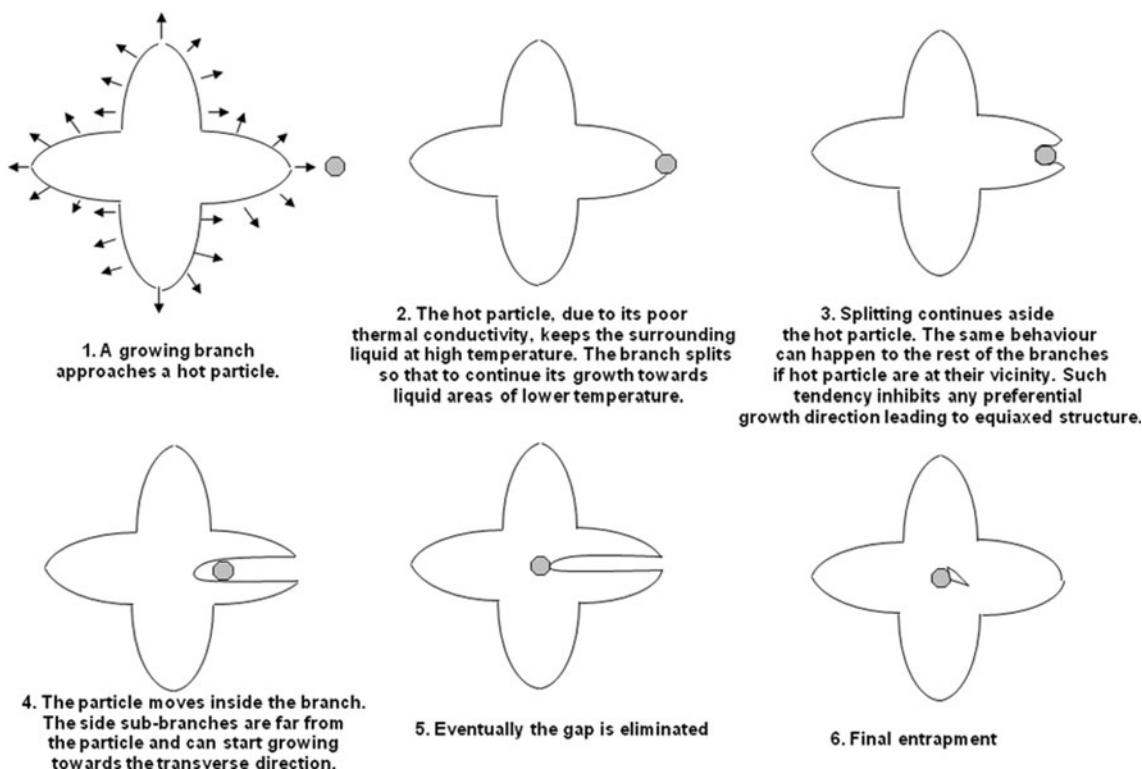


Fig. 4 Mechanism of particle entrapment by the primary α -dendrites. The first three steps are also responsible for the equiaxed morphology

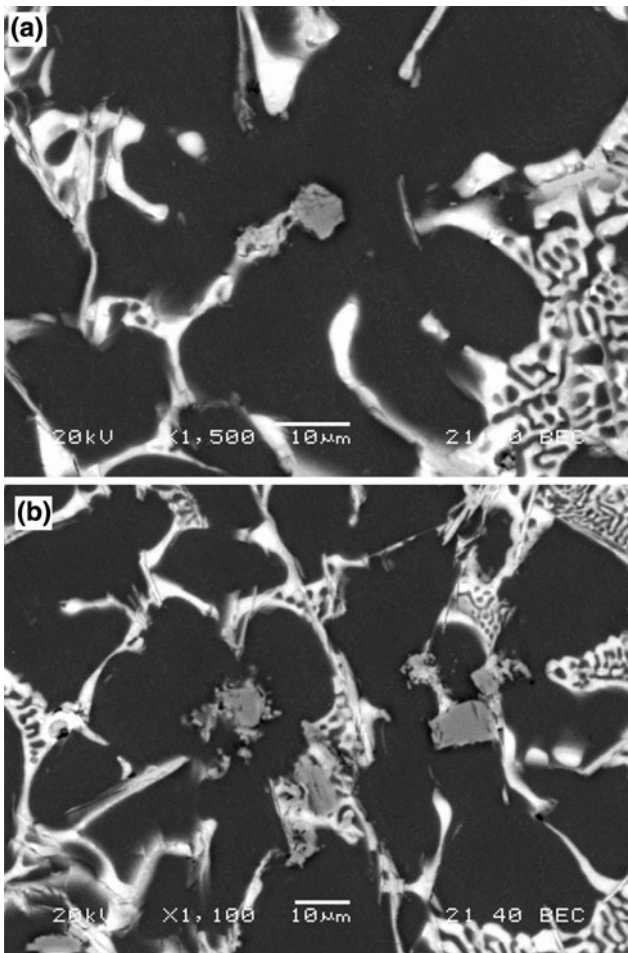


Fig. 5 SEM pictures (back-scattered mode) showing the last two steps of particle entrapment: (a) extensive dendrite arm splitting and (b) side branches converging and final entrapment

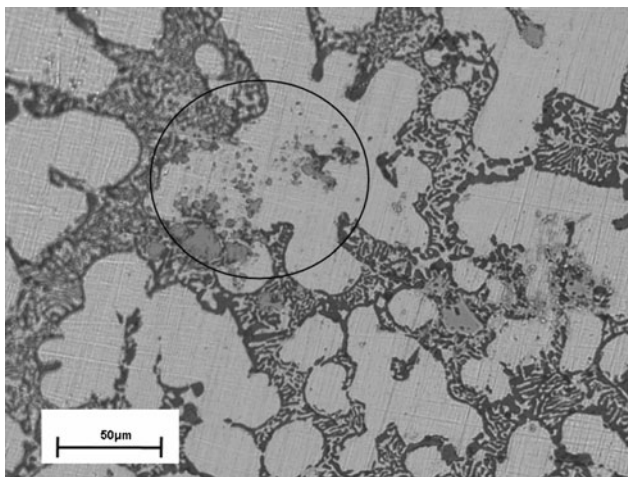


Fig. 6 Optical micrograph showing the existence of needle-like intermetallics associated with the presence of TiC particles. EDX analysis revealed stoichiometries close to $\text{Cu}_9\text{Al}_4\text{Ni}$ type

interdendritic areas. However, in Fig. 6, it can be observed that smaller particles are rather engulfed within the primary dendrites than entrapped or pushed at the interdendritic areas.

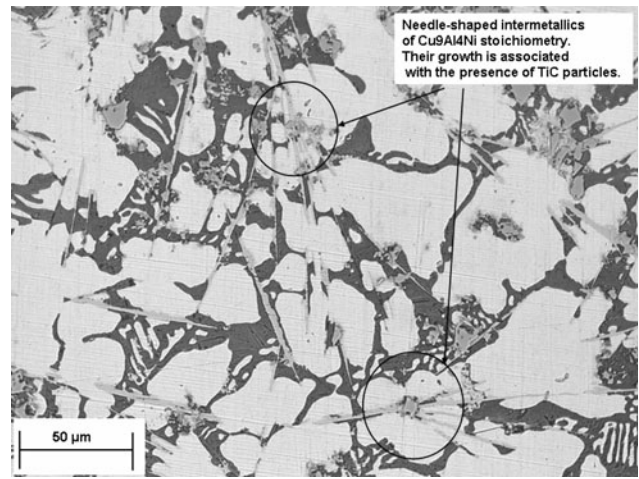


Fig. 7 Optical micrograph showing the engulfment of smaller particles by the advancing dendrite tips

According to the models predicting the particle behavior in front of an advanced solidification front, the large particles should be engulfed. A possible explanation would be as follows: small particles possess less actual heat content than the large ones. During solidification, the large particles, due to their high thermal content, cannot follow the temperature drop, remain hot and behave in a manner previously described either by being pushed or by splitting the dendrite tips. For the small particles, however, due to their low thermal content, it would be easier to follow the temperature decrease of the melt and by the time the solidification front reaches them their temperature is at such levels that they can be engulfed by it. Such particle behavior may be an indication that the models to predict particle-front interaction which take into account the thermal conductivities of the involved phases should be reformed in such a way that they take into account their actual thermal content.

Another interesting point to mention is the location of intermetallic phases and their association with the presence of the TiC particles in the case of the $\text{Al}_{20}\text{Cu}_1\text{Ni}$ matrix material. As shown in Fig. 7, the intermetallic phases are located at the interdendritic regions and there is significant evidence that their nucleation is associated with the ceramic particle surface, meaning that they have acted as heterogeneous nucleation sites. EDX analysis of the intermetallic phase showed stoichiometries of the $\text{Cu}_9\text{Al}_4\text{Ni}$ type.

4. Conclusions

- An Al-20 vol.% TiC master composite was diluted in Al7Si, Al₂₀Cu, and Al₂₀CuNi alloy matrices resulting in the production of 3 to 5 vol.% TiC composite materials.
- The good wetting characteristics between TiC particles and the matrix, ensured by the use of halide salts during the manufacturing stage, are responsible for the successful particle insertion.
- The preferential placement of TiC particles at the grain boundaries of the primary α -phase, which present equiaxed morphology, is attributed to particle pushing

phenomena by the solidification front. The differences in thermal conductivities and the solidification front critical velocity concept explain this behavior.

- The presence of smaller particles engulfed by the primary α -phase may suggest the reconsideration of the particle-solidification front interaction models.
- The formation of intermetallic phases is associated with the presence of TiC particles.

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