EFFECT OF TIC POWDER ADDITION ON THE GRAIN REFINEMENT RESPONSE OF B319 ALUMINIUM ALLOY

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Abstract

In this research, efficiency of grain refinement of permanent mold cast B319 aluminum alloy using titanium carbide (TiC) grain refiner was investigated. The grain refiner was added at 0.031, 0.05 and 0.1 wt%. In-situ solidification analysis was carried out, along with microsctructure and mechanical testing characterization. The results of cooling curve analysis suggest that addition of TiC delayed the time to reach the solidus temperature, increased the liquidus temperature, and decreased the amount of undercooling. Scanning Electron Microscopy (SEM) and chemical analysis revealed that Ti was present in solution uniformly throughout the matrix.

Introduction

Aluminum alloy B319 is a popular variant from the family of Al-7Si aluminum alloys currently used for automotive cylinder block and head components due to its optimal creep resistance, high temperature dimensional stability and corrosion resistance. B319, being a hypoeutectic alloy, solidifies by first forming a large α -Al phase, followed by second phases and eutectics. When the α -Al retains its fine and equiaxed morphology, the resulting as-cast mechanical strength, fluidity and feeding characteristics of the casting significantly improve [1]. However, in order to promote and control formation of the fine equiaxed α -Al morphology, Al-5Ti-1B master alloys are traditionally used as grain refiners. These grain refiners introduce TiAl₃ and TiB₂ substrates into the liquid metal and provide heterogeneous nucleation sites for the formation of α -Al grains [1-3]

Sigworth and Guzowski [4] have suggested that in the grain refinement of Al-Si alloys with titanium, if more than 2wt% Si is present in solution, a layer of titanium silicide forms on inoculating particles and reduces their grain refinement efficiency. A crystallographic study by Qiu [5] on the orientation relationship of nucleant sites TiAl3, TiB2 with Al and Si confirmed that formation of a titanium silicide layer of Ti₅Si₃ was the most likely cause of grain refinement poisoning. Oiu also demonstrated that TiAl₃ particles were more susceptible to the formation of the titanium silicide layer as compared to TiB₂, because of reasonably good crystallographic interfacial matching with Ti₅Si₃. This approach was later expanded by experimental work where a better grain refining of Al-7Si alloys refined with boron (B) was achieved with lower amounts of titanium silicide [1,2]. However, according to duplex nucleation theory [6], TiB₂ particles alone are ineffective until covered by a laver of TiAl₃.

Recent efforts have led to the development of Al-Ti-C based grain refiners, which introduce titanium carbide (TiC) particles as heterogeneous nucleating sites. The 'carbide' theory postulated by Cibula [7] suggests that TiC particles directly nucleate α -Al,

because of their excellent lattice matching. Extensive work [3,7-10] supports this theory, since Ti- and C-rich crystals in grain refined aluminum grains were observed. In the work of Kumar on grain refinement of Al-7Si alloys subject to slow cooling rates [3], addition of 0.1 wt% of Al-5Ti-1.2C master alloy was seen to grain refine as efficiently as 1wt% of Al-3B or Al-Ti-3B master alloys. Kumar also reported that with higher TiC addition levels, coarser grains were observed because of agglomeration of TiC particles. This agglomeration supports previous studies of Mohanty and Gruzleski [11], which suggests that TiC particles are unstable in the Al-7Si melt and dissolve within ~30 minutes of holding time. However, at lower addition levels, TiC provides better grain refinement than Al-Ti-B or Al-B grain refiners. Yet, TiC based grain refiners, in contrast to the Al-Ti-B type, have not been established in industry as a potential grain refiners due to the difficulty in preparing Al-Ti-C master alloys. For example, formation of carbides of Al₄C₃ and Ti₃AlC₅ during processing of the master alloy might decrease the grain refining potency of Al-Ti-C particles [12]. Also, homogeneous introduction of Ti-C particles into the melt remains a challenge.

Thus, this study focused on studying the effect of adding micron-sized TiC particles into liquid B319 aluminum alloys with the view of grain refinement.

Experiment Details

Titanium Carbide (TiC) powder with a 3µm particle size was added at three addition levels to B319 aluminum alloy during permanent mold casting. Based on the work with Al-5Ti-1.2C [12], optimum addition level of TiC was expected to be at 0.031 wt%. Thus, casting trials were carried out with 0, 0.031, 0.05 and 0.1 wt% TiC addition levels. To avoid TiC agglomeration during processing, the total mass of powder was separated into four equal amounts, and individual quarters were wrapped in aluminum packets. For all casting trials, 2kg of B319 alloy were melted in an electric resistance furnace and heated to820°C, at which point



Figure 1: Representative casting

aluminum packets with TiC powder were introduced into the melt and pushed below liquid metal surface. Then, the melt was slowly stirred using a mechanical stirrer for 1 min and held at temperature for 2 min. Thereafter, the melt was skimmed and cooled to 775°C and poured into a preheated (350 °C) permanent mold. Figure 1 shows picture of a complete casting upon ejection from the permanent mold.

Thermocouples placed in the casting cavity were used to record in-situ casting solidification. Also, casting sections were polished using standard procedures for Aluminum alloys [13] and the microstructure was studied using Zeiss AxioVert A1m optical microscope with Buehler Image Analysis software. Also, a Tescan Mira³ Scanning Electron Microscope with X-ray Dispersive Spectrometer (X-MAX) were used for high-resolution imaging and chemical analysis, respectively.

Results and Discussions

Effect of TiC on microstructure:

Figure 2(a-d) shows the grain size of the cast samples of B319 alloy as cast and grain refined with different TiC addition levels. The figure shows that addition of TiC powder changed the volume fraction of the secondary phase present in the microstructure and the maximum was observed at 0.1wt% TiC.

Figure 3 shows the grain size vs addition level of TiC. Grain size reduces from $23.69 \mu m$ to $20.70 \mu m$ on adding 0.031 wt% TiC that corresponds to ~13% reduction in grain size. However, grain size increased with higher addition levels, which is in agreement with a previous study on TiC grain refiners [12]

Effect of TiC addition on alloy solidification:

The effect of TiC addition on the solidification path between the liquidus point and end of solidification for the B319 alloy is provided in Figure 4. Since all casting process parameters remained constant, the difference in the solidification path has been attributed to TiC addition. The results suggest that addition of TiC has delayed the onset of precipitation of intermetallics forming below 560°C which, in turn delayed the onset of eutectic reaction. The extension of the freezing range was also observed as an enhanced fluidity of the liquid metal during mold filling.

The results also suggest that grain refinement postponed dendrite coherency due to which dendrite impingement occurred at higher solid fraction (leading to enhanced metal fluidity) [14], which is also consistent with previous studies that suggest addition of grain refiner results in higher fluidity and better feeding characteristics [15,16]



Figure 2: Microstructure of B319 alloy grain refined with a) No addition, b) 0.031wt% TiC, c) 0.05wt% TiC and d) 0.1wt% TiC at 100X magnification



Figure 3: Grain size of B319 alloy grain refined vs addition level of TiC

The solidification characteristics were also examined by examining the difference between T_u (undercooling temperature) and T_m (maximum temperature of α -Al growth). In general, the value of T_u decreased with enhanced grain refinement [17]. This trend was observed in the present research as well.

Figure 5 shows the change in T_u and ΔT vs. addition level of TiC. It is evident from the graph that addition of 0.031wt% of TiC powder resulted in a decrease of both T_u and ΔT with later being zero in this case. In contrast, with higher addition level of TiC, the T_u and ΔT increased in comparison to 0.031 wt%.



Figure 4: Cooling curves of B319 alloy for different TiC addition level

According to the stoichiometry of TiC, 0.031 wt% of TiC corresponds to 0.025 wt% of Ti, which is approximately 0.5 wt% of Al-5Ti-1.2C master alloy that has been reported as an optimum addition level in a previous study [12]. Addition of TiC above this level was seen to result in grain coarsening. As can be seen in Table 1, the relative increase in T_u and ΔT of the present work for castings with 0.05 and 1.0 wt% TiC also supports this observation.

Table 1: Variation of T_u (undercooling temperature), T_m (Maximum temperature of a-Al growth) and AT with addition level of TiC

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ſ	Temperature	No	0.031 wt%	0.05 wt%	0.1 wt%
	of interest	Addition	TiC	TiC	TiC
ſ	Tm	602.79	598.50	602.21	600.81
	Tu	602.32	598.50	601.60	600.67
	ΔT	0.47	0	0.61	0.14



Figure 5: Tu (undercooling temperature) and ΔT for B319 alloy grain refined with different addition level of TiC

General discussion:

The addition of TiC was seen to shift the liquidus curve of the B319 alloy for all addition levels. This shift (or delay in attaining eutectic point) affected the phase transformations occurring in the semisolid range, where important intermetallics



Figure 6: SEM micrographs of B319 alloy grain refined with 0.031 wt% TiC addition, a) BSE image and XRD Map for b) Carbon and d) Ti distribution



Figure 7: SEM-XEDS images of a) SEM micrograph showing the linescan across secondary phase b) XRD data for the corresponding line scan

such as CuAl₂ and MgSi₂ form. Due to the extension of the freezing range, diffusion of solute from solid α -Al phase through diffusion boundary layer was possible, and enabled enhanced solute segregation. XRD maps (fig. 6) reveal that Ti was uniformly distributed throughout the matrix, suggesting that Ti was likely acting as a growth restrictor, rather than a nucleating particle for α -Al.

Growth restriction would enhance solute enrichment of the liquid phase during casting solidification, which would ultimately influence grain growth via the "growth restriction theory" as suggested by [18]. Examination of the microstructure of the present castings suggests that the addition of TiC increased the fraction of secondary phases (indicative of solute segregation). Specifically, in the alloy with 0.1wt% TiC addition, the area fraction of secondary phases was greatest, as was seen in Figure 2 SEM analysis of the α -Al revealed that the diffusional boundary layer in their vicinity has reduced with increasing TiC addition level. A decrease in the diffusional boundary layer is indicative of higher diffusion rates through the matrix, which explains the better solute segregation observed with TiC addition.

The composition of the dominant secondary phase was consistent with that of $CuAl_2$, as seen from the XEDS linescans shown in figure 7. Thus, it appears that addition of TiC influenced the volume fraction of precipitated $CuAl_2$ phase, due to growth restriction and solute enrichment in liquid during solidification.

Conclusion

Based on the results performed in this study, the following conclusions may be postulated:

a) Addition of TiC powder directly, instead in the form of a master alloy with Al, increases the fluidity and feeding

characteristics.

- *b)* Cooling curves shifts to the right and results in delayed attainment of solidus temperature.
- c) Fraction of secondary phase, CuAl2, increases with increase in addition level of TiC in the melt.
- Addition of TiC powder increases the growth restriction of solidifying α-Al.
- *e)* Ti is distributed evenly in the solid solution, and not acting as an active nucleation site.

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