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THEORY AND PRACTICE OF GRAIN REFINING FOR ALUMINIUM ALLOYS - UTILIZING COVERAL MTS 1582



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The need for smaller grains is vital to achieving the required properties when pouring most cast aluminum alloys. Whether the desired results are high mechanical properties, leaker free castings, a cosmetic appearance or improved structural soundness, smaller grains are impactfully beneficial. Accordingly, there is a desire to improve both grain refining and the ability to quickly and effectively assess grain refinement effectiveness. This paper discusses both the need for smaller grains and the principle fundamentals of grain refining. Moreover, the paper reviews commercially-available grain refiner forms and currently available methods for assessing grain refinement. Finally, the paper introduces a new and improved flux form grain refiner (COVERAL MTS 1582) and documents two recently successful case studies where the COVERAL MTS 1582 was utilized to improve castings in both a low pressure wheel foundry and a high production sand moulding foundry, respectively.



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INTRODUCTION

Grain refining is an essential part of the aluminium casting process which aims at reducing the size of primary aluminium grains during the solidification phase. This process has many benefits for most hypoeutectic aluminium alloys as it improves feeding, elongation and mechanical properties, increases resistance to fatigue, improves casting machinability, reduces hot tears, helps disperse micro-shrinkage, decreases the size of porosities and reduces thermal treatment cycles. Historically, grain refinement has been achieved using master alloys, with the most commonly used grain refiner mechanism involving the release of Titanium diboride into the melt. Grain refining is especially important in aluminium foundries using investment, sand, gravity die, or low pressure die casting processes due to the potential for delayed cooling and complex casting designs with varying section thickness.

In general, those castings with slower cooling rates and larger variation in casting thickness, require grain refinement more than other casting designs.

There are several casting segments where grain refining is critical including:

- + Wheel foundries where grain refinement and cleaning are crucial for achieving the required feeding and cosmetic surface finished of the casting.
- + Safety critical automotive castings such as suspension parts, turbochargers, and brake components which require good fatigue properties.
- + General automotive castings like cylinder heads, engine blocks, manifolds in gravity diecast where an intermediate level of grain refinement might suffice for the mechanical property requirements, but the improved feeding from grain refinement helps prevent leakers.

- + Aerospace and military castings requiring high mechanical properties for difficult applications, grain refining is highly beneficial.
- + Sand and investment castings where the long solidification times cause large grain growth and difficult feed paths without optimized grain refining.

GRAIN REFINEMENT MECHANISM IN ALUMINIUM ALLOYS

TARGET OF ALL MELT TREATMENT PROCEDURES IS AN IMPROVEMENT OF MECHANICAL PROPERTIES

Grain refinement affects the α -mixed crystal in the alloy. At decreasing temperature those α -mixed crystals grow. Grain size depends on cooling rate during solidification. The addition or formation of nuclei increases solidification speed and decreases the grain size.

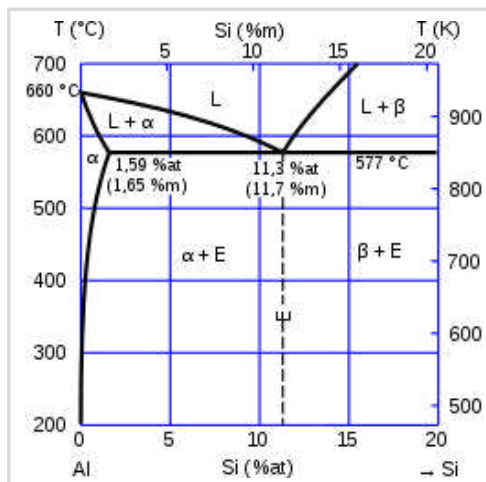


Figure 1: Al-Si phase diagram

Ceramic	Angle
TiB ₂	60°
ZrB ₂	106°
HfB ₂	134°
TaB ₂	125°
TiC	118°
ZrC	150°
SiC	135°
HfC	148°
NbC	136°
TaC	145°
TiN	135°
ZrN	167°
NbN	156°
AlN	138°

Table 1: Contact angle of different ceramic materials [1]

MASTER ALLOY AND CHEMICAL PRODUCTS COMPARISON

Considerations when using master alloy grain refiners

- + AlTi5B1 - AlTi3B1 - AlTi5B0,2 - AlTi10B1
- + TiB₂ nuclei are pre-formed in an aluminium matrix
- + Easy to apply
- + Risk of oxides or impurities in the rod or waffle
- + Moisture and oxides on rod surface contaminate melt

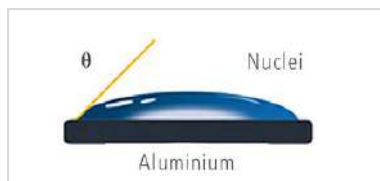


Figure 2: Nuclei needs a good wettability by melt

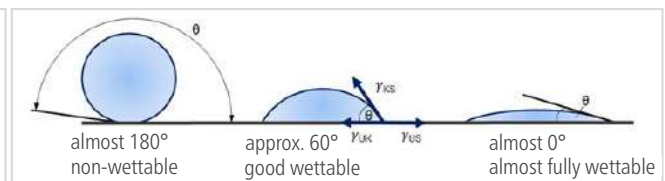


Figure 3: Heterogeneous nucleation as a function of wetting angle

$$\sigma_{\alpha F} = \sigma_{\beta F} + \sigma_{\alpha\beta} \cos(\theta)$$

$\sigma_{\alpha F}$ = melt surface energy
 $\sigma_{\beta F}$ = surface energy of nuclei
 $\sigma_{\alpha\beta}$ = interface energy between nuclei and melt

Figure 4: Young's equation

Benefits of chemical products are:

- + Contain metallic titanium and boron salt or titanium and boron salt
- + TiB₂ nuclei are in-situ formed in the melt – fresh surface – higher surface energy and lower θ angle
- + No risk of impurities
- + Additional cleaning effect



REASONS FOR BETTER MECHANICAL STRENGTH WITH CHEMICAL PRODUCTS

We identified several reasons for achieving better mechanical strength with chemical products which are:

- + Chemical products and master alloys with pre-formed nuclei impact the contact angle θ differently
- + θ for $TiB_2 = 60^\circ$ is a theoretical value for an ideal nucleus
- + θ for TiB_2 from master alloys is significantly higher due to reduced surface energy
- + θ for TiB_2 from chemical products is close to 60° or even below due to fluxing effect from chemicals (fluorides)

COVERAL MTS 1582

FOSECO has developed a novel granulated flux COVERAL MTS 1582 that is capable of both grain refining and cleaning aluminium alloy melts. COVERAL MTS 1582 is highly concentrated in titanium and boron which form both titanium diboride and aluminum boride in situ leaving fresh nuclei within the aluminium melt. These finely dispersed species are highly efficient nuclei that promote a fine equiaxed grain growth during solidification.

In addition to strong grain refining, COVERAL MTS 1582 flux is also a very good cleaning product that will react to remove oxides and inclusions from the melt. No additional cleaning/drossing flux is required, resulting in lower overall process costs. COVERAL MTS 1582 is a sodium- and calcium-free granulated flux suitable for all types of aluminium alloys except hyper-eutectic alloys but including those alloys containing large amounts of magnesium.

APPLICATION OF COVERAL MTS 1582

COVERAL MTS 1582 is specially designed for use with FOSECO'S MTS 1500 rotary degassing and melt treatment equipment, whereby controlled flux additions are made directly into a melt vortex and mixed vigorously. PLC controlled additions of the treatment flux are added into the vortex and mixed to complete reaction prior to the vortex breaker baffle board re-engaging the melt, effectively stopping the vortex. After the vortex has been stopped, the MTS completes a standard rotary degassing process and the treated metal in the ladle or crucible is used for transferring and/or casting.

For further information of the MTS 1500 process, readers are advised to review Foundry Practice Issue 247 (2007) or the Foundry Practice Special Edition for AFS CastExpo (2008).

Both issues feature excellent articles on the MTS 1500 technology [References 2 and 3].

MTS 1582 should be used with the melt at a temperature higher than 720 C. The reaction by-product from this treatment produces an extremely dry ash-like dross that is easily separated from the liquid metal with a coated skimmer or similar tool.

EVALUATING GRAIN REFINEMENT EFFECTIVENESS

Since grain refinement is critical to achieve the desired properties of aluminium castings, it is important that there are methods for assessing grain refinement effectiveness. The most common methods for evaluating grain refinement effectiveness are as follows:

- + Elemental spectroscopy
- + Thermal analysis
- + Microstructural evaluation

ELEMENTAL SPECTROSCOPY

Elemental spectroscopy is perhaps the most commonly employed method for assessing grain refinement, but it is also the least effective of the three methods listed. Spectroscopy only determines the total concentration of an element - however Titanium is usually present in other forms and phases in addition to TiB_2 and these other phases do not impact grain structure. Foundries will measure Ti into the alloy range (typically 0.10-0.25% by weight) and assume that because they are in range, they are achieving sufficient grain refinement. Consequently, given this issue, some foundries will also measure boron (typical range 5-25ppm) as an additional control. Tight controls of Ti and B do typically result in effective grain refining; however, more advanced methods like thermal analysis and microstructural analysis ensure higher probabilities of optimized grain refinement.

THERMAL ANALYSIS

Thermal Analysis is perhaps the fastest growing method for assessing grain refinement as it is quick and more accurate than elemental spectroscopy. The THERMATEST* 5000 NG III (pictured in Figure 5) is a widely used thermal analysis unit used to quickly and accurately assess grain refinement effectiveness in aluminium alloys. Thermal analysis involves collecting data of temperature versus time of a solidifying melt sample and comparing the curve to a set of known reference curves algorithmically. The THERMATEST 5000 NG III unit's algorithm analyzes the sample curve liquidus and computes a score on a scale from 1-9 for evaluating grain fineness (GF). A score of 1 references a curve that compares with curves exhibiting no grain refining.

In contrast, a GF score of 9 is achieved when the sample curve compares with those curves known to have produced "perfect" grain refining of melts with the same alloy composition. A pictorial representation of the THERMATEST 5000 NG III grain refinement levels is provided in Figure 7. Of note, THERMATEST 5000 NG III unit also provides the side benefit of helping to assess eutectic modification effectiveness in Al-Si alloys [References 4 and 5].

EVALUATION OF GRAIN SIZE WITH THERMAL ANALYSIS

For a given cooling speed, the size of the grain depends on the amplitude and duration of the undercooling, which appears at the formation of primary aluminium crystals.

- + When the undercooling is high and duration medium (Fig 6a), grain size is coarse.
- + When there is no undercooling (Fig 6b), grain size is very fine.
- + When undercooling is low but duration is high, the grain size is very coarse.



Figure 5: Photograph of a THERMATEST 5000 NG III unit

THERMATEST 5000 NG III measures the following Liquidus parameters:

- + Temperature θ_2 (°C)
- + Undercooling $\Delta\theta$ (°C)
- + The duration of undercooling t_1 (in seconds)

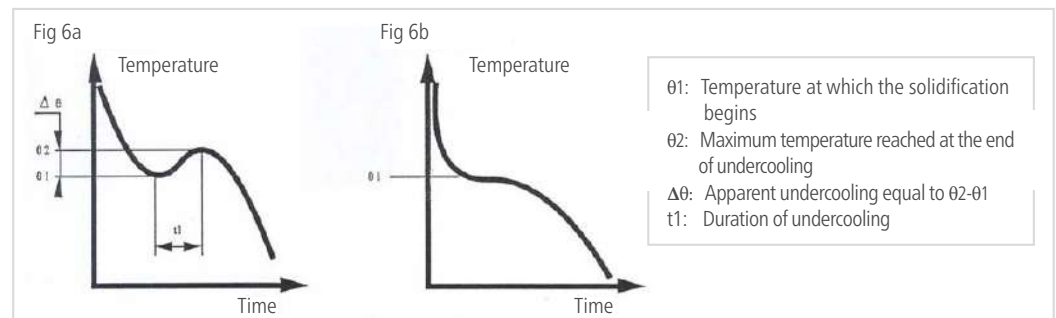


Figure 6a and b: Profiles of the cooling curve at the solidification of primary aluminium crystals in case of hypoeutectic alloy

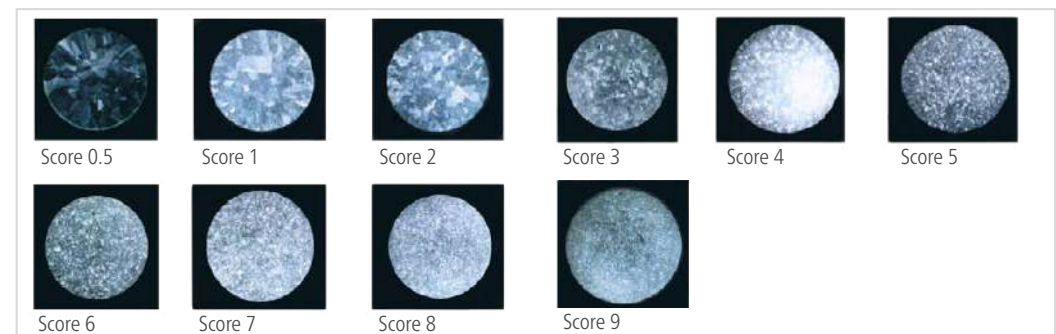


Figure 7: Test of grain refinement - Standard plate with Grain Fineness (GF)

Grain refinement is considered fully optimized when the undercooling is nil and grain size index is equal to 9. However, for certain alloys and thin shaped castings in permanent moulds, a lower grain size index (5 - 9) would be expected and is acceptable due to the higher cooling rate with permanent die casting.

We recommend setting a minimum grain size index for each casting, correlated with desired elongation of mechanical properties. For Al-Cu5%MgTi alloys, the absence of undercooling may not be sufficient to avoid hot tears. A stronger grain refinement is recommended to improve the alloy's performance.



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LIQUIDUS CURVES: COMPARISON OF TiB RODS WITH COVERAL MTS 1582

The lower the undercooling at Liquidus, the stronger the grain refinement. COVERAL MTS 1582, at much lower addition rate (0.11 % vs 0.2 % for AlTi5B1 rods), performs better compared to AlTi5B1 rods.

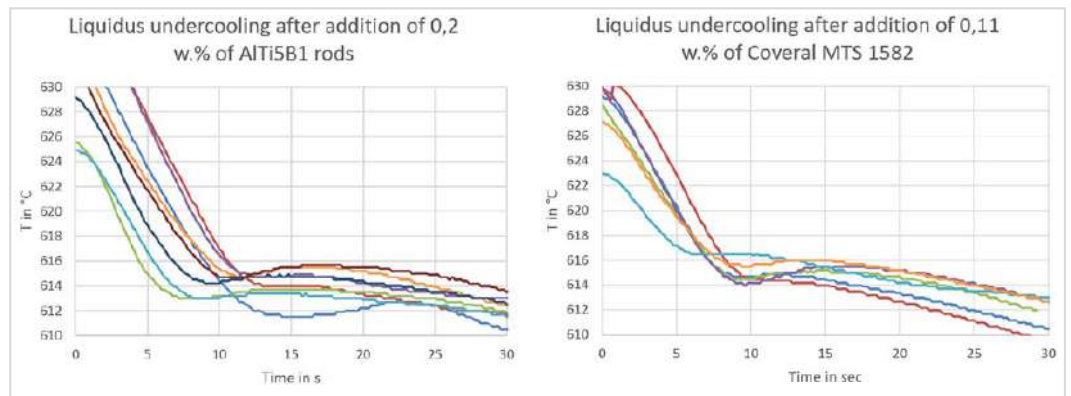


Figure 8: Thermal analysis curves

OPTICAL MICROSCOPY (BARKER TEST)

Optical microscopy is the final methodology employed by foundries to assess grain refinement. Optical microscopy is considered the most representative method for assessing grain refinement but is time-consuming and resource intensive. Optical microscopy involves grinding and polishing test specimens to microscopic levels to be evaluated for grain size under a microscope. One popular method for optical microscopy is the Barker test. The LectroPol-5 from Struers is used for electrolytic etching with Barker reagent consisting of a 5% tetrafluoroboric acid in distilled water. The sample to be tested acts as an anode in a galvanic cell, which removes material from the sample surface and an anodic layer can be formed. With the Barker method, under a polarized light, a colored representation of the grain structure of aluminum materials is achieved. It is possible to carry out microscopic testing with up to 1000x magnification.

Alloy: AlSi7Mg0,3
COVERAL MTS 1582
Addition rate: 0,1%



Figure 9a: Before treatment. Grain size d_m [μm] = 984



Figure 9b: After treatment. Grain size d_m [μm] = 206



Figure 10: Comparison of TiB rods with Coveral MTS 1582: grain size



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CASE STUDIES

WITH COVERAL MTS 1582

1: European foundry

A European wheel foundry was interested in improving its melt treatment practices by utilizing COVERAL MTS 1582 with a FDU featuring MTS 1500 technology. This wheel foundry pours a standard AISi7Mg alloy and historically performed grain refining by making manual additions of TiBor rod into a transfer ladle during degassing. It was the foundry's target to automate the grain refining process all while capturing the typical benefits (drier dross, lower spend, smaller grain) achieved when grain refining with COVERAL MTS 1582. The treatment parameters of the new process featuring COVERAL MTS 1582 can be found in Table 2.

After the new process grain refining with COVERAL MTS 1582 was implemented, pictures were taken of the ladle dross (Figure 11), thermal analysis curves (Figure 13) and microstructures (Figure 12).

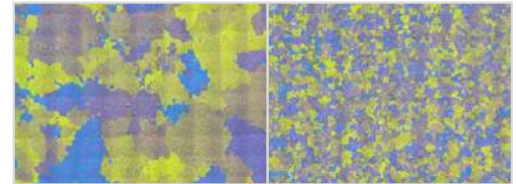


Figure 12: Microstructure before and after treatment with COVERAL MTS 1582.

Treatment parameters	
Ladle	INSURAL ATL 600 with 500 kg of AISi7Mg
Temperature	730 - 760 °C
Addition rate	250 g COVERAL MTS 1582 (0.05 % of the melt weight)
Treatment time	6 minutes
Inert gas flow	20 l/min N ₂
Rotor speed	450 rpm for MTS FDR 190.70

Table 2: European Wheel Foundry (EWF) treatment parameters.



Figure 11: Photograph of extremely dry dross in transfer ladle after treatment with COVERAL MTS 1582.

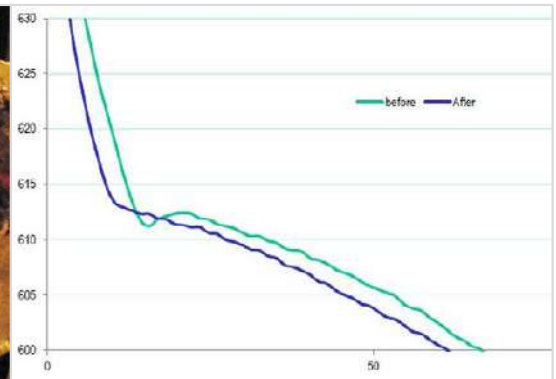


Figure 13: Thermal analysis curves

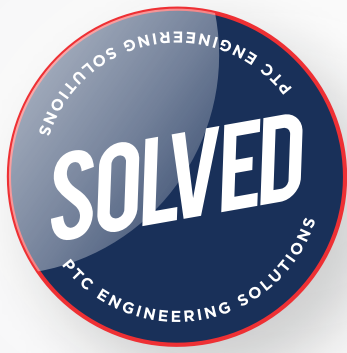
2: American foundry

Littlestown Foundry is a sand and low-pressure (LP) mould aluminium foundry in Littlestown, Pennsylvania in the USA. The main alloy poured by Littlestown Foundry is a standard 356 alloy (AISi7Mg). In the sand foundry, Littlestown casts some difficult castings that are subjected to pressure testing with air to make sure they are leak free for application. After reducing scrap through improved grain refining in the LP foundry from 13.6% to 2.7% by converting from metallic TiBor (10%Ti, 1%B) to COVERAL MTS 1582, a similar project was undertaken in the sand foundry. The aim was that by improving the grain refining using COVERAL MTS 1582 - introduced via an MTS 1500 unit - in place of metallic TiBor rod, the sand foundry would see similar benefits in the form of reduced leakers and lower spend.

The first part of the project involved using a THERMATEST 5000 NG III unit to assess the incumbent procedure and then developing an optimized procedure using the MTS 1500 and COVERAL MTS 1582. The results of the THERMATEST 5000 NG III evaluation are presented in Table 3.

Sample #	Average Grain Fineness (GF)
Sample before treatments	5.8
Standard TiBor Additions	6.8
COVERAL MTS 1582	9.0

Table 3: Results of THERMATEST 5000 NG III evaluation with the COVERAL MTS 1582 grain refining flux.



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The THERMATEST 5000 NG III evaluation confirmed that the metallic TiBor rod was successful in raising the Grain Fineness value from insufficient (5.8/9.0) to an improved and more acceptable level of grain refining (6.8/9.0). However, the THERMATEST 5000 NG III unit also confirmed that a huge improvement to a fully optimized level of perfect grain refinement (9.0/9.0) was possible with the COVERAL MTS 1582. Hence, mechanical test bars were poured and evaluated to assess any potential impact of the new process featuring the COVERAL MTS 1582.

The results of the mechanical testing evaluation are shown in Table 4. The results exhibited positive improvement in all three metrics evaluated, i.e., ultimate tensile strength (UTS), yield strength (YS) and % Elongation. Accordingly, the decision was made to convert to the new process to make a full assessment of the new process featuring COVERAL MTS 1582 and a FDU featuring MTS 1500 technology.

Test	Incumbent TiBor Process	New Process Featuring MTS 1500 & COVERAL MTS 1582
UTS in psi (MPa)	40,000 (276)	41,290 (285)
YS in psi (MPa)	34,500 (238)	35,100 (242)
Elongation (%)	4%	5%

Table 4: Results of mechanical testing of preceding treatment samples and samples collected after the implementation of a MTS 1500 with COVERAL MTS 1582.

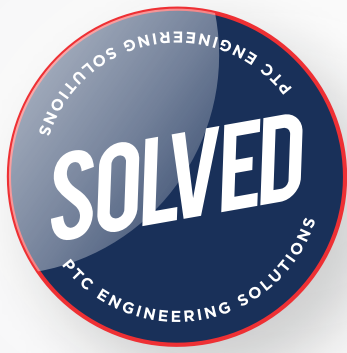
Finally, after four months in production, the new process change was evaluated economically. The following economic benefits were achieved after implementation:

- + Reduction in annual projected spend on grain refiners and cleaning flux by \$276 per day, \$1,380 per week, \$5,750 per month or more than \$69,000 per year.
- + A ten-fold reduction in projected impregnation costs from a starting point that exceeds \$1,500 per month to less than \$150 per month.
- + The calculated payback for the MTS 1500 unit when factoring in the lower grain refining spend, the lower flux cleaning flux spend and offsetting it with the slightly higher spend on filters is just over 6 months.

A full peer-reviewed paper (paper #19-015) on the Littlestown Case Study was published with the AFS 123rd Metalcasting Congress Proceedings in April 2019 and is available for a more extensive review.

SUMMARY & CONCLUSIONS

COVERAL MTS 1582 is a universal grain refining and cleaning flux for treating aluminium alloys. It forms in situ Aluminium boride and Titanium boride which are the most suitable nuclei, within aluminium melts. Creating TiB₂ nuclei in situ is more effective than releasing pre-made TiB₂ nuclei into a melt. Elemental spectroscopy, thermal analysis with a THERMATEST 5000 NG III and optical microscopy are three methods for assessing grain refinement effectiveness within a melt; the latter two methods being the most efficient. Experience in both a low pressure wheel foundry and high production greensand foundry has confirmed the benefits of superior casting mechanical properties and lower overall process costs when grain refining using COVERAL MTS 1582 through an MTS 1500 unit.



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