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**DIFFICULTIES IN GRAIN REFINING ALUMINUM LITHIUM ALLOYS USING
COMMERCIAL Al-Ti AND Al-Ti-Bor MASTER ALLOYS**

L.C. LABARRE, R.S. JAMES, J.J. WITTERS, R.J. O'MALLEY and
M.R. EMPAGE

*Aluminum Company of America, Alcoa Technical Center, Alcoa
Center, PA 15069, U.S.A.*

Abstract

High strength aluminum alloys, especially those containing zirconium, are historically difficult to grain refine. Early efforts by Alcoa indicated that the addition of lithium to these alloys increases difficulty with grain refinement. Although grain refining of alloys 2090 and 2091 was possible, consistent results were not obtained. Statistically-designed experiments were used to quickly identify critical parameters in both the casting and grain refining operations. Responses from variables including alloy composition, amount and type of grain refining addition, melt temperature and cooling rate were quantified prior to establishing control limits. Production ingot cast by Alcoa are now consistently grain refined.

Future work will simultaneously investigate the impact of important process variables on the fundamental mechanisms of grain refining and establish appropriate targets and ranges for these variables.

The Problem

Aluminum-Lithium alloys were found to be more difficult to grain refine than conventional aluminum alloys. Standard grain-refining practices for aluminum alloys yielded inconsistent results as illustrated in Figure 1. The presence of zirconium in the alloy explained some of this difficulty, but the effects of lithium on grain refining were unclear. Typical problems included TCG (twinned columnar grains) and non-uniform grain size.

These problems in grain refining led to processing and product quality difficulties. Detrimental effects were noted in the areas of:

1. Casting
2. Property degradation
3. Fabricability

During casting the presence of TCG caused increased cracking due to hot tearing. This necessitated a reduction in the casting rate to avoid cracking, thus increasing the overall product cost.

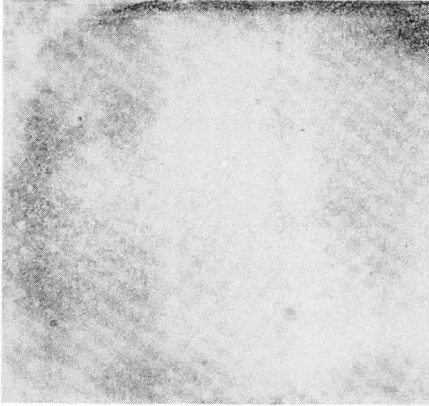
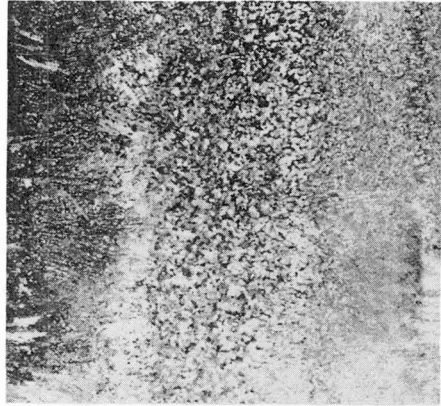
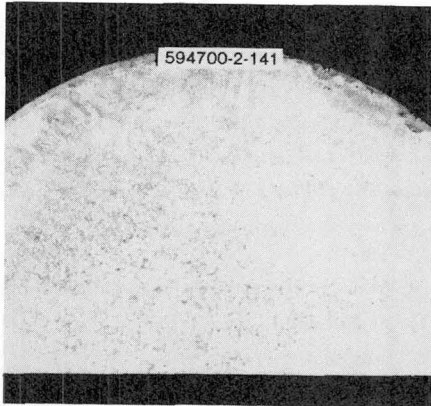
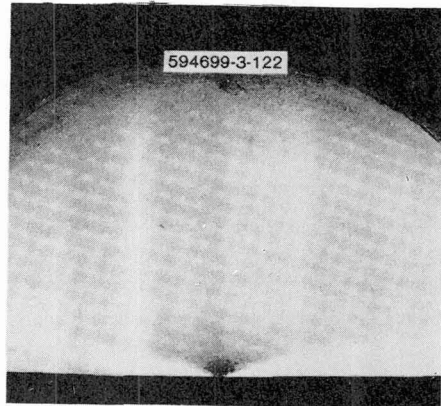
**Fig. 1A****Fig. 1B****Fig. 1C****Fig. 1D**

Figure 1: Inconsistent grain refining in Al-Li alloys using similar practices. Note TCG in Figure 1B and inconsistent grain size in Figures 1C and 1D.

Degradation of aluminum-lithium alloys due to non-uniform grain refining occurred in two property areas. First, TCG was found to decrease elongation as shown in Figure 2. Secondly, the as-cast grain size strongly correlated with final product toughness. Fine as-cast grain size consistently resulted in lower product toughness (Figure 3). Although the mechanisms of this degradation are not yet understood, the interactions between as-cast grain size and product properties are being pursued in order to identify the preferred grain size.

Fabrication difficulties with ingots containing TCG included surface checking on the rolling face of slabs and cracking of the slabs during hot rolling.

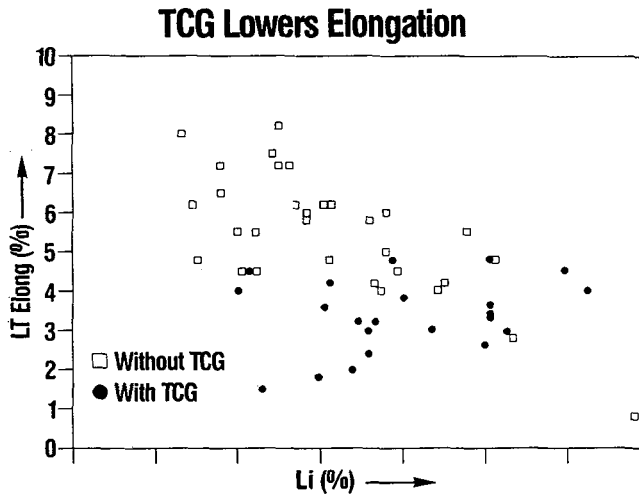


Figure 2: TCG decreased elongation.

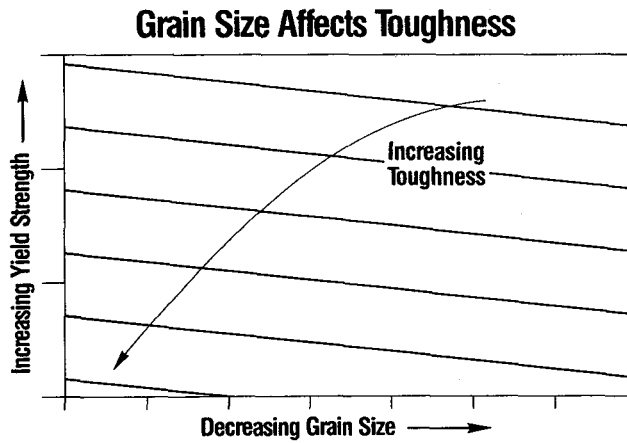


Figure 3: A fine as-cast grain size inexplicably caused lower final product toughness.

The Objectives

The production of consistently grain-refined products at minimum cost is the main objective of this study. The following sub-objectives were defined in order to meet this overall goal:

1. Identify critical process parameters which determine ingot grain size and the presence of TCG.
2. Determine the preferred grain size for product properties.
3. Determine process region boundaries wherein preferred grain size is attainable.
4. Determine process region boundaries wherein TCG is eliminated.
5. Set process targets and operating limits based upon process response regions and alloy composition.
6. Develop a control scheme to insure that process targets and operating limits are met consistently.

The Experimental Plan

In order to quickly fulfill the first objective listed above, a statistically designed experiment was selected to determine both the critical and non-critical operating variables, as well as significant interactions between these variables. Before the experiment could actually be run, potentially influential operating variables and the responses of interest had to be determined. The responses were easily identified as grain size and the presence of TCG.

Operating variables were selected based upon:

- review of scientific theory of grain refining
- interviews with technical experts
- analysis of historical data
- cross-analysis of production control charts

The resultant selection of eight operating variables for investigation led to the design of the 2^{8-4} fractional factorial experiment shown in Table 1. The high and low levels for each variable were selected to reflect process/alloy extremes.

This approach was selected because of its many potential benefits. First, utilization of a statistically-designed experiment minimized the confounding effects of production noise. It also enabled the identification of critical variables early in the product's life cycle and prior to the establishment of a long production history. Once the effect of operating variables are determined, their processing ranges can be set according to product sensitivity.

In addition to improving product consistency, both cost and manufacturing benefits are realized from the control of critical variables. Costly control loops and data acquisition are minimized to the bare essentials. Process flexibility is maintained by tightening operating ranges only as is necessary. This eliminates potential conflicts with required ranges for other process steps.

FRACTION FACTORIAL DESIGN FOR AL-LI GRAIN REFINING INVESTIGATION

Run Order	<u>Variable</u>							
	Ti	B	Temp	Li	Zr	Mg	Method	Coolant
1	+	-	-	+	+	-	+	-
2	-	-	-	-	-	-	-	-
3	-	-	+	+	-	-	+	+
4	+	+	-	+	-	-	-	+
5	-	+	-	-	+	-	+	+
6	+	-	+	-	+	-	-	+
7	+	+	+	-	-	-	+	-
8	-	+	+	+	+	-	-	-
9	-	-	+	-	+	+	+	-
10	+	+	+	+	+	+	+	+
11	+	-	+	+	-	+	-	-
12	+	+	-	-	+	+	-	-
13	-	-	-	+	+	+	-	+
14	+	-	-	-	-	+	+	+
15	-	+	-	+	-	+	+	-
16	-	+	+	-	-	+	-	+

Table 1: Experimental Outline

Experimental Procedures

The above experiment was run according to the following procedures:

Operator Instructions

For each of the sixteen runs:

- Alloy
- Flux
- Analyze composition
- Adjust (if necessary)
- Grain refining sample of baseline metal
- Add grain refiner rod
- Stir for 45 seconds

- Take grain refining samples at the following residence times:
 1. 3 minutes
 2. 10 minutes
 3. 20 minutes
 4. 35 minutes
 5. 60 minutes
 6. 120 minutes
- Cut all seven samples in half and etch to reveal grain structure

The ALCOA standard grain refining test mold was modified in order to obtain thermal profiles. These profiles permit future analysis of thermal gradients and their effects on grain size and TCG. The experimental setup is shown in Figure 4.

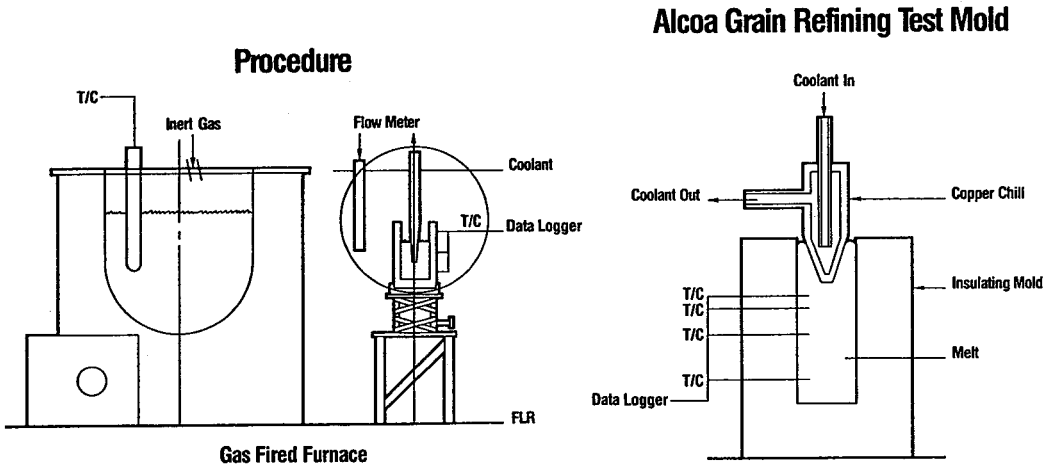


Figure 4: Experimental Grain Refining Test Mold Setup

Results

The above experiment yielded a wide range of grain refinement with resultant variations in both grain size and TCG. In order to analyze the data statistically, a ranking system was established which assigned a quantitative rating to each structure. This system, shown in Figure 5, assigned higher point values to samples with increasing degrees of refinement.

The effects of the operating variables, defined as the average at the upper level of the factor minus the average at its lower level, were evaluated and plotted as a function of residence time. The criticality of each variable was then determined based upon the magnitude of its effect. The distribution of these effects are shown in Table 2.

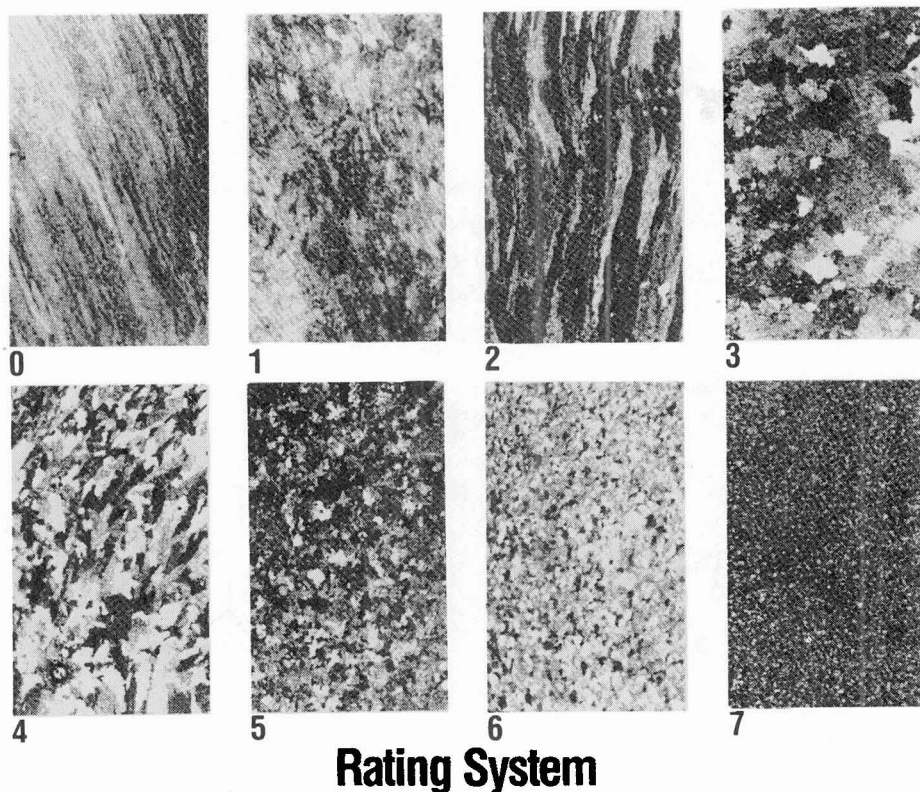


Figure 5

DISTRIBUTION OF VARIABLE EFFECTS

Overall Effect or Change from Baseline

Critical: $Effect > 1$ Less-Critical: $Effect \sim 1$ Insignificant: $Effect < 1$

Critical	Less-Critical	Insignificant
<i>Ti</i>	<i>B</i>	<i>Zr</i> after addition
<i>Mg</i>	<i>Temp</i>	<i>Li</i> after addition
<i>Zr</i> in baseline	<i>Method</i>	(<i>Temp·Mg</i>)
<i>Li</i> in baseline	<i>Coolant</i>	(<i>Temp·Zr</i>)
		(<i>Temp·Coolant</i>)
		(<i>Temp·Li</i>)

Table 2: Results showing the relative criticality of the operating variables examined.

INTERACTIVE EFFECTS

+		0
[(Ti·Temp)+(B·Method)]	[(Li·Mg)+(Zr·Coolant)]	Zr)+(Mg·Coolant)]
[(B·Temp)+(Ti·Method)]	[(Ti·Li)+(B·Coolant)+(Zr·Method)]	
[(Ti·B)+(Temp·Method)]	[(B·Li)+(Ti·Coolant)+(Mg·Method)]	
[(Li·Coolant)+(Zr·Mg)]	[(B·Zr)+(Ti·Mg)+(Method·Coolant)]	
	[(Ti·Zr)+(B·Mg)+(Li·Method)]	

Table 3: Results showing the sum of the effects of confounded two-factor interactions.

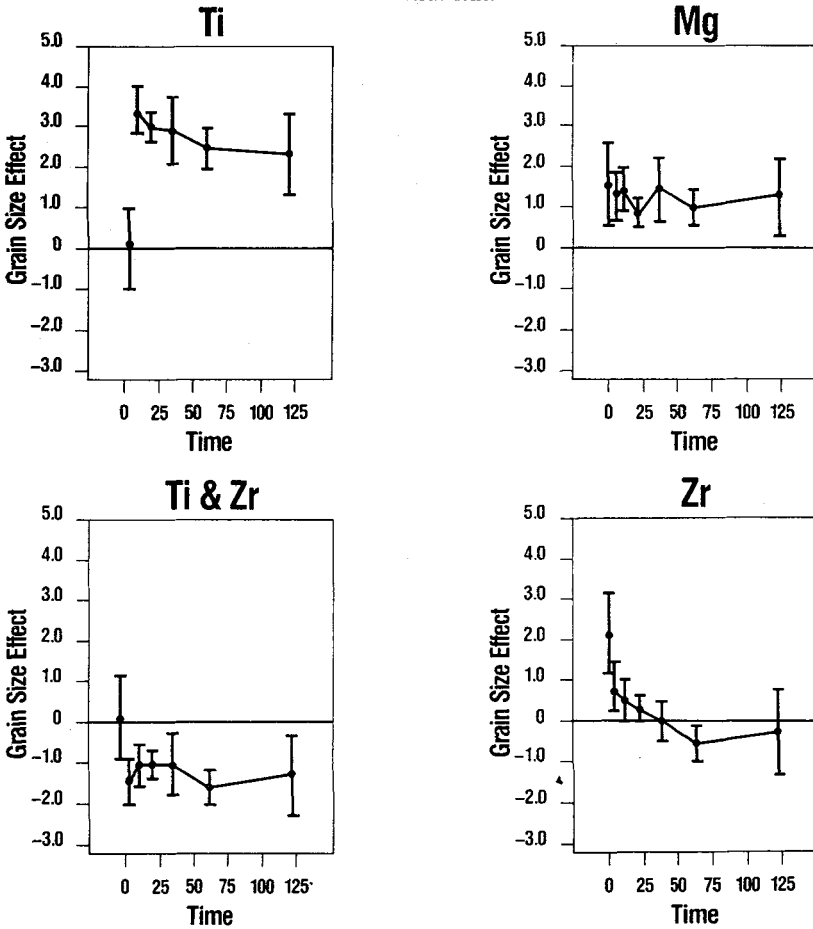


Figure 6: Variable Effect vs. Time

The 2^{8-4} fractional factorial experiment permitted evaluation of all main, single-variable effects. Main effects are confounded only with third and higher-order interactions. The two-factor interactions are all mutually confounded in groups of four. For this reason, the effect of an individual two-factor interaction cannot be easily determined. However, in cases where the baseline effect differs from the effect at positive times, the interactive terms can be separated into two categories: those present before the grain refiner addition and, those occurring after. When only a single two-factor interaction falls into either category, its effect can be determined. Thus, four such interactive effects were identified as insignificant in Table 2. All other interactions remain in groups. Although the sign and magnitude of individual effects within a group cannot be determined, the sum of the effects can be stated as either positive, negative or zero as listed in Table 3.

Plots of the estimate of effect as a function of residence time are shown in Figure 6 for some of the critical operating variables and interactions. The approximate 95% confidence limits are plotted as error bars for each value.

Conclusions

The strong positive effect of magnesium indicates that the optimum practice for grain refining will differ for alloys 2090 and 2091. Titanium was identified as the single most influential main effect, thereby requiring the greatest control. Other main effects, such as boron and temperature, were shown to be less critical; however, their overall contribution to grain refining cannot be fully determined until the magnitude of their interactive effects is discerned. Further experimentation is thus required to unconfound the remaining two-factor interactions.

Of the eight operating variables examined, only titanium, boron and method are unique to the grain refining step of the ingot manufacturing process. All other variables impact other functions. It is, therefore, advantageous to control grain refining through tight control of the "three-function specific" variables while maintaining flexibility around the others. The identification of titanium as a key operating variable demonstrates the feasibility for this type of process control.

Future Work

Future work will be aimed in three directions:

1. Fundamental understanding
2. Process control
3. Wrought product improvement

In continuing our pursuit of understanding the fundamentals of our experiments, we intend to:

- Examine the possible mechanisms in light of classical grain refinement theory.
- Compare the results with conventional aluminum alloys.

In the area of process control, we have set the following objectives:

1. Verify off-line results on production ingot.
2. Determine the sign and magnitude of effects from individual two-factor interactions where appropriate.

3. Investigate potential difference between residual and added titanium/boride for scrap loop considerations.
4. Determine process region boundaries for preferred grain size without TCG.
5. Determine the interactions between critical variables for grain refinement with those of other processing steps.
6. Set process targets and operating limits .
7. Develop a cost-effective control scheme.

Our future work in wrought products will include:

- Evaluation of wrought product properties as a function of grain size and grain refining mechanism.
- Determine preferred grain size.

Overall, it is our intention to encourage the use of these techniques in other processing steps, such as:

- Identify critical variables for each function.
- Develop compatible and cost-effective control loops.
- Produce a consistently high quality product.