

# Research Programs

## Optimization of Heat Treatment Using Fluidized Bed (FB) Reactors

### Research Team:

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### Objectives

The goal of this project was to develop the fundamental knowledge base for heat- treating of aluminum components using fluidized bed (FB) reactors. Specifically, we studied the kinetics of phase transformation and microstructural evolution during post solidification heat treatment of cast aluminum components through FB processing. The microstructure and properties of cast aluminum components that are heat-treated in FB were compared to their counterparts that were conventionally heat-treated. Within the scope of this project we assessed the economical benefits and environmental impact of FB reactors.

### Project Overview

Heat treatment characteristics of T4 (solutionizing, quenching, and natural aging) and T6 (solutionizing, quenching, and artificial aging) tempered cast alloys were studied. Castings were heat treated using fluidized bed (FB). For comparative purposes, castings were also heat treated using conventional furnace (CF). In case of SSM A357 alloy, the casting was heat treated to T5 temper (artificial aging) using both FB, and CF. Detailed microstructural characterizations were carried out using both optical and scanning electron microscopy. Microstructural evolution during various stages of heat treatments such as solutionizing, quenching, and aging were analyzed and studied with the use of thermal analyses. Finally, tensile properties were correlated with their resultant microstructures.

The project was divided into three phases. These are:

- Phase I - Optimize solution heat treatment parameters of cast Al alloys using FB
- Phase II - Optimize aging temperature and time of cast Al alloys using FB
- Phase III - Evaluate quench sensitivity of FB

This project commenced in April 2002 and ended in May, 2005; the project was funded by the ACRC consortium members. Several important research findings were found, which are pertinent to both the scientific and industrial communities. The primary objective of this project was to develop a fundamental understanding of fluidized bed heat treating of Al castings and optimize their processing parameters. Some salient features of fluidized beds are: 1) high heating rate, 2) uniform heating of castings, 3) excellent temperature uniformity within the bed, and 4) environmentally friendly. These advantages were partly realized during the past three years through extensive experiments and analysis on heat treating of various cast alloys. Various castings selected for this study, can be grouped into two major categories. These are

1. Al-Si-Mg (A356-with low Mg content (~0.35%), and D357-with high Mg content (~0.6%), and
2. Al-Si-Cu-Mg (354 alloy-with low Cu content (~1.81%), and 319 type alloy-with high copper content (~3%).

These alloys were all grain refined and also modified with Sr. In case of D357 alloy, the heat treatment characteristics of both unmodified and Sr modified alloys were studied. These alloys are commonly used in industry for various automotive applications and were chosen for this study because of the strong interest and support from our consortium members. In addition to conventional casting (i.e. liquid state), some selected studies were also made on heat treatment characteristics of semi-solid cast A357 alloy.

## **Outcome / Impact**

Some salient conclusions for each alloy studied in this project are given below:

### **A356 alloy - T4 and T6 temper**

- The heating rate in FB is almost an order of magnitude higher than in conventional furnaces (CF). Components take about the same time (~4 min.) to reach the solutionizing and aging temperatures in FB (heating time is independent of final temperature). However, through CF, the time to reach the aging temperature is twice that for reaching the solutionizing temperature. This is due to the heating mechanism in FB reactor, which is forced heat convection through solid particles (when a small thermal mass is introduced into a large thermal mass of the fluidized bed).
- Solutionizing in FB reactor results in faster spheroidization of eutectic Si particles vis-à-vis conventional furnace. At 1000°F (538°C) and 1030°F (554°C) solutionizing temperatures, Si particles get significantly spheroidized in 30 min and 15 min, respectively in a FB reactor as compared to about 90 min using CF.
- Solutionizing at 1030°F results in transformation of the Fe rich phase from  $p\text{-Al}_8\text{Mg}_3\text{FeSi}_6$  to  $b\text{-Al}_3\text{FeSi}$ , whereas at 1005°F (541.6°C) this transformation does not occur. It takes about 1 to 2 hours to complete the transformation of  $\pi$ -phase to  $\beta$ -phase in FB at 1030°F.
- The precipitation kinetics of  $\text{Mg}_2\text{Si}$  particles is faster in the FB reactor than in CF. Aging of FB solutionized samples (at 1000°F for 30 min) results in small spherical  $\text{Mg}_2\text{Si}$  precipitates of 30-100 nm size range, whereas long CF solutionized samples (1000°F for 9 hours) age to yield coarse  $\text{Mg}_2\text{Si}$  needle-like precipitates of 1000nm-2000nm size range.
- The tensile strength of the A356 alloy solutionized using FB at 1020°F (549°C) is greater than at 1030°F. This is due to the transformation of  $\pi$  phase to  $\beta$  phase on solutionizing at 1030°F.
- Optimum solutionizing time (T4 temper) for FB processing is in the range of 15-30 minutes.
- Total heat treatment time for T6 temper using a FB reactor is less than 2 hours.

### **D357 alloy - T4 and T6 temper**

- The high heating rate in the FB results in greater spheroidization kinetics of eutectic Si during solution heat treatment than in CF.
- Mechanism of Si fragmentation is through brittle fracture due to thermal mismatch between Al and Si and rapid neck formation in the FB heat-treated castings. In contrast to this, the mechanism of Si fragmentation in alloy solution heat-treated using a CF is through relatively slow neck formation and its propagation.
- Optimum solution heat treatment times using the FB for unmodified and Sr modified alloy are 60 minutes, and 30 minutes, respectively.
- Four minutes are required to attain the aging temperature in the FB (fluidized bed), compared to sixty minutes in the CF (conventional furnace); over an order of magnitude difference.
- Nucleation rate of  $\text{Mg}_2\text{Si}$  particles in the FB is greater than that in the CF.
- Thermal analysis shows an endothermic reaction during aging in the CF. No such transformation was observed during aging of the alloy using the FB.
- Nucleation rate of  $\text{Mg}_2\text{Si}$  particles is greater in the Sr modified alloy as compared to the unmodified alloy.

- Coarsening rate of  $Mg_2Si$  particles is greater in the unmodified alloy as compared to the Sr modified alloy. Coalescence of smaller particles is the dominant mechanism for coarsening of  $Mg_2Si$  particles in the unmodified alloy. Whereas in the Sr modified alloy, Ostwald ripening is the dominant mechanism of coarsening.
- Optimum aging time, in the FB is in the order of an hour. Total heat treatment cycle for T6 temper in Al-Si-Mg alloys is reduced to less than 2 hours using the FB; a significant reduction of cycle time.

### **354 and 319 alloys - T4 and T6 temper**

- Significant spherodization of eutectic Si in 354, and 319 alloys is achieved within 30 minutes of solution heat treatment using the FB.
- The  $Al_3Mg_8Cu_2Si_6$  phase dissolved completely within 45 minutes in both 354, and 319 alloy during solution heat treatment using the FB.
- The  $Mg_2Si$  phase dissolves completely within 45 minutes of solution heat treatment in Al matrix of 319 alloy using the FB.
- No significant effect on morphologies of  $Al_2Cu$  on solution heat treatment within 120 minutes using the FB is observed. Even prolonged solution heat treatment for 360 minutes using the CF does not lead to complete dissolution of  $Al_2Cu$  phase.
- No significant change on the morphology of Fe rich intermetallics within 120 minutes of solution heat treatment using the FB is observed. On prolonged solution heat treatment for 360 minutes using the CF, a reduction in angularity is observed. However, the reduction of angularity does not play any significant role on their tensile properties.
- The optimum solution heat treatment time for 354, and 319 alloy is 45 minutes. Beyond which no significant change in the tensile properties (UTS, YS, and elongation) is observed.
- $Al_3Cu_2Mg_8Si_6$ , and  $Mg_2Si$  particles precipitate during aging in the matrix of the 354, and 319 type alloy.
- The coalescence of smaller particles is the dominant mechanism for coarsening of  $Al_3Cu_2Mg_8Si_6$  particles in both 354, and 319 type alloy.
- The precipitation and coarsening rate of  $Al_3Cu_2Mg_8Si_6$  particles is greater when using the FB vis-à-vis the CF.
- Aging of both 354, and 319 alloys at 392°F (200°C) results to greater strength (UTS and YS) as compared to those aged at 464°F (240°C).
- Aging (T6 treatment) decreases elongation of both 354, and 319 alloys.
- Using FB, the aging time can be reduced to about an hour as compared to 5 hours of aging time using CF.
- Total heat treatment time for T6 temper is less than 2 hours.

### **Quench sensitivity (D357, 354, and 319 alloys) - T4 and T6 temper**

- The cooling rate of the casting during quenching in the FB is lower than those in the water.
- The change in cooling rate ( $\Delta(dT/dt)$ ) was more drastic during quenching in water as compared to those in the FB.
- The thermal analysis result shows that the FB quenching of 354 alloy results in the formation of some metastable phases; whereas no such transformations were observed during water quenching of the same.
- Quenching alloys (D357, 354, and 319) in the FB followed by natural aging (T4 temper) yields greater UTS, and YS vis-à-vis water quenched alloys. In contrast, elongations of alloys quenched in the FB are lower than those quenched in water.
- Both UTS and YS values of T6 tempered D357 alloys (unmodified and Sr modified) are greater when quenched in water as compared to those quenched in the FB. On contrary, in case of Al-Si-Mg-Cu alloys (354, and 319) no significant difference in UTS and YS values are observed between castings aged after quenching in the FB and water.

- Elongation of the FB quenched Sr modified alloy treated to T6 temper was greater than those quenched in water. In contrast, elongation in the FB quenched Al-Si-Mg-Cu alloys (354, and 319) treated to T6 temper are lower than those quenched in the FB.
- Al-Si-Mg alloys are more quench sensitive than Al-Si-Mg-Cu alloys. This is because the simulated TTT diagrams predict the formation of greater percent of GP zones in Al-Si-Mg-Cu alloys, whereas in Al-Si-Mg alloys, GP zones do not form. These GP zone are heterogeneous nucleation sites for precipitation and enhances aging kinetics.

### A357 SSM alloy - T5 temper

- The precipitation kinetics of Mg<sub>2</sub>Si particles is faster in the FB reactor than in CF.
- Aging using a FB reactor for shorter interval of times (1-3 hours) results in small spherical Mg<sub>2</sub>Si precipitates of 30-100 nm in size. In contrast to this, components aged using CF for longer time (5 hours) reveal both spherical and needle like particles of length of 1000 nm-2000 nm.
- Microhardness measurements reveal that the optimum aging time of thixocast A357 alloy at 300°F (149°C), 350°F (177°C), and 400°F (204°C) are 180 min., 150 min., and 60 min., respectively.
- The functional test results show that through fluidized bed processing, the aging time can be reduced to an hour, which is significantly less compared to conventional air convective furnace aging, where it takes about 5 hours to achieve similar strength.

### Publications

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2. M. M. Makhlof, D. Apelian, S.K. Chaudhury and C. Bergman, " Heat treatment of aluminium cast components in fluidized bed", in proceedings of the **1st International Light Metals Technology Conference**, Brisbane, Australia, 18-20 September, 2003, Edited by A. Dahle, P. 371-376.
3. S.K. Chaudhury, S. Shankar, D. Apelian, and J. Vanwert, "Short Cycle Heat Treating with Fluidized Beds: Structure Evolution", in proceeding of **International Conference on Structural Aluminum Castings**, Orlando, Florida, USA, November 2-4, 2003.
4. S.K. Chaudhury, L. Wang, and D. Apelian, "Fluidized Bed Reactor Heat Treatment of A356 Alloy: Microstructure Analysis and Mechanical Properties", **AFS Transactions**, 112, 04-055, 2004.
5. S.K. Chaudhury, Q. Pan, J. Heimsch, and D. Apelian, "Response of semi-solid processed Al based cast alloy during fluidized bed heat treatment", in proceedings of **8<sup>th</sup> International Conference on Semi Solid Processing of Metals and Alloys**, Limassol, Cyprus, 21-23 September, 2004, published by NADCA, Wheeling, Illinois.
6. D. Apelian and S.K. Chaudhury, "Fluidized bed heat treatment of aluminum cast components", **Journal De Physique IV**, December, 120, p. 555-562, 2004.
7. S.K. Chaudhury, D. Apelian, "Fluidized Bed Heat Treatment of Cast Al Alloys", in **Shape Casting: The John Campbell Symposium**, published by TMS - ISBN # 0-87339-583-2, pp 283-293 (2005).
8. S.K. Chaudhury and D. Apelian, "Effects of solution heat treatment on microstructure and mechanical properties of Al-Si-Cu-Mg (354) alloy using a fluidized bed reactor", **AFS Transactions**, 113, 05-071, 2005.
9. D. Apelian and S.K. Chaudhury, "Heat treatment of Aluminum cast components-Recent developments and future challenges", **World Foundry Organization Congress**, St. Louis, MO, USA, April, 2005.
10. S.K. Chaudhury and D. Apelian, "Effect of rapid heating on solutionizing characteristics of Al-Si-Mg alloy using a Fluidized bed", **Metallurgical and Materials Transaction A**, 37A, March, p. 763-778, 2006.

11. S.K. Chaudhury and D. Apeilan, "Fluidized bed heat treatment of cast Al-Si-Mg-Cu alloys", ***Metallurgical and Materials Transactions A***, 37A, p. 2295-2313, 2006.
12. S.K. Chaudhury and D. Apeilan, "Effect of rapid heating on aging characteristics of T6 tempered Al-Si-Mg alloy using a Fluidized bed", ***Journal of Materials Science***, **41**, p. 4684-4690, 2006.
13. S.K. Chaudhury and D. Apeilan, "Effects of fluidized bed quenching on heat treating characteristics of cast Al-Si-Mg and Al-Si-Mg-Cu alloys", ***International Journal of Cast Metals Research*** (in press), 2006.

## Fluidized Bed - An Innovative Method for Heat Treating Al Casting

### Objectives

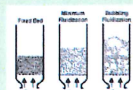
- Develop the fundamental knowledge base for heat treating of Al castings using FB,
- Establish microstructure-property correlation of FB heat-treated castings.

### Challenges

- Reduce heat treating cost
- Obtain uniform microstructure and consistent mechanical properties
- Minimize part distortion due to heat treatment
- Lower residual stress
- Increase fatigue strength

### Background

#### Principle of Fluidized Bed



Increasing gas flow rate

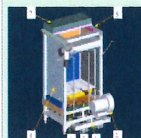
- Fluidization involves feeding gas through a bed of fine particles (typically sand)
- The moving gas separates the particles enough that they slide freely past each other
- The bed reacts similar to a fluid

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### Abstract

Improved mechanical properties in cast Al alloys are often achieved through heat treatment comprising of solution heat treating, quenching, and aging, successively. With impetus to be cost effective, it is imperative to reduce the long heat treating times needed (i.e. up to 15-20 hours) without any reduction in performance. Fluidized beds (FB) provide an attractive heat treating technology for components with more efficient energy transfer and thereby reducing the net heat treatment time and enhance productivity. Here, we review the fundamentals of FBs, highlight their advantages and examine application of FBs to heat treat cast Al alloys. Mechanical properties data along with the resultant microstructure are presented. Using FBs, the optimum solutionizing, and aging time for cast Al alloys are reduced to 30-45 min, and 60 min, respectively. The high heating rate in FB enhances both solutionizing, and precipitation kinetics. In addition, FBs can be used to quench alloys (which are less quench sensitive) in lieu of water as it significantly reduces both residual stress and distortion in components.

### Fluidized Bed (FB)



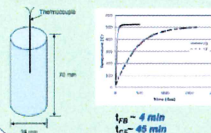
**Applications**  
Heat treatment (HT) of alloys  
- Solutionize  
- Age  
- Quench

#### Advantages

- Rapid and uniform heating
- Potential for reducing heat treat heating time
- Reduce temperature gradient within parts
- Reduce residual stress
- Lower risk of parts distortion and cracking
- Low maintenance cost
- Clean and safe operation

### Comparison of Heat Transfer Coefficients

Heating Medium	$h_{\text{conv}}$ ( $\text{Wm}^{-2}\text{K}^{-1}$ )
Lead	1200-1800
Salt bath	900-1200
Fluidized bed	500-700
Radiant fluidized bed	200-300
Forced circulation furnace	150-300
Radiant atmosphere furnace	120-220
Vacuum furnace	120-200



### Effect of Solution HT on Eutectic Si



> Spheroidization kinetics in FB > CF

#### Hypothesis

- FB kinetics → high thermal stresses at eutectic Si/Al interface
- Why? → thermal mismatch - eutectic Si and eutectic  $\alpha$ -Al
- Coefficient of thermal expansion  
Si (3.61  $\mu\text{m/m}^\circ\text{C}$ )  
Al (25.5  $\mu\text{m/m}^\circ\text{C}$ )

### Mechanism of Si Fragmentation in FB

#### Solution HT



- Si fragmentation in FB → brittle fracture and neck formation and its propagation, whereas in CF → neck formation and propagation
- Kinetics of neck formation and its propagation in FB > CF, due to the generation of thermal stresses on eutectic Si caused by thermal expansion
- Si particles at periphery of eutectic region are more prone to brittle failure as compared to that inside eutectic in FB HT

### Tensile Properties - T4

Alloy/Temp.	As-cast	UTS (Kai)	CF (time)
A356/538°C	24.51	38.15 (30)	38.53 (350)
D357/543°C	24.39	41.4 (30)	43.86(360)
354/527°C	29.61	40.88 (45)	40.05(360)
319/493°C	35.56	44.68 (45)	42.5(360)

Quenched in water at 25°C; Incubation time = 48 hours

> Optimum Solutionizing time in FB = 30-45 min.

### Effect of Aging at 170°C- Al-7Si-0.35Mg

FB-60 min. CF-300 min.



Solutionized in FB @538°C/60 min

Quenched in water -aged in FB

Quenched in water -aged in CF

Fast precipitation

Non-uniform distribution of Mg<sub>2</sub>Si particles

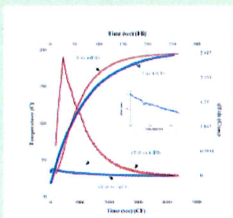
Spherical particles: 30-100nm

Slow precipitation

Non-uniform distribution of Mg<sub>2</sub>Si particles

Size of needle: 100nm-300nm

### Thermal Analysis

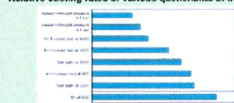


#### Hypothesis

Slow heating rate in CF results in endothermic transformations due to the dissolution of GP zones / Clusters → decrease heterogeneous sites for nucleation → Reduce precipitation; On contrary there was no endothermic transformation for alloys aged in FB.

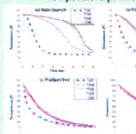
### Application of FB as Quenchant

#### Relative cooling rates of various quenchants at the center of specimen\*

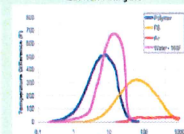


\* For P.W. Reformation, Heat treatment in FB furnace, WPI, Andover, MA, USA

#### Temperature profile

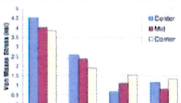


#### Quench Analysis



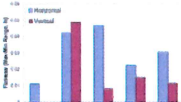
>  $\Delta(T/dt)_{\text{water quench}} > \Delta(T/dt)_{\text{FB quench}} \Rightarrow$  less residual stress and distortion in casting quenched in FB

### Residual Stress Analysis



> Residual stress in FB < water

### Distortion Analysis



> Distortion in FB < Water

### Highlights

- FB - an emerging technology with light temperature control and high heating rate
- Microstructural benefits using FB
  - Solutionizing: Greater coarsening and spheroidizing rate of Si
  - Aging: Greater nucleation and coarsening rate of precipitates
  - Quenching: Reduce residual stress, and distortion
- Reduce Heat treatment time using FB
  - Optimum time
    - ~30-45 min. for solution heat treatment
    - ~Less than 2 hours for T6 temper

### Ongoing work ...

- Study the effect of FB heat treatment on fatigue performance of Al Castings
- Validation of hypothesis on dissolution of GP zones / clusters during aging - Using LEAP (Local Electrode Atom Probe)
- Tailor quench sensitivity of Al-Si-Mg cast alloys