

PNNL-35668

Overcoming the Barriers to Lightweighting by Enabling Low-Cost and High-Performance Structural Automotive Aluminum Castings (CRADA 404)

September 2021

Mert Efe Tarang Mungole David Weiss Aashish Rohatgi



Prepared for the U.S. Department of Energy under Contract DE-AC05-76RL01830

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Pacific Northwest National Laboratory Richland, Washington 99354

Cooperative Research and Development Agreement (CRADA) Final Report

30th September 2021

In accordance with Requirements set forth in the terms of the CRADA, this document is the CRADA Final Report, including a list of Subject Inventions, to be provided to PNNL Information Release who will forward to the DOE Office of Scientific and Technical Information as part of the commitment to the public to demonstrate results of federally funded research.

Parties to the Agreement: Pacific Northwest National Laboratory and Eck Industries, Inc. CRADA number: 404

CRADA Title: Overcoming the Barriers to Lightweighting by Enabling Low-Cost and High-Performance Structural Automotive Aluminum Castings

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DOE Program Office: EERE-Vehicle Technologies Office

Joint Work Statement Funding Table showing DOE funding commitment:

Funding	Project	Project	Project	Project	Project	
	Year 1	Year 2	Year 3	Year 4	Year 5	TOTALS
Government						
DOE						
Other						
Total Govt.	\$125,000	\$125,000				\$250,000
Participant						
In-Kind	\$125,000	\$125,000				
Funds-In						
FAC						
Total Participant	\$125,000	\$125,000				\$250,000
TOTAL	\$250,000	\$250,000				\$500,000
CRADA						
Value						

This product contains Protected CRADA Information, which was produced on 30th Sept. 2021 under CRADA No. 404 and is not to be further disclosed for a period of five (5) years from the date it was produced except as expressly provided for in the CRADA.

Executive Summary

The purpose of this program between PNNL and Eck Industries, Inc. (Eck) is to lower the cost and improve the performance of aluminum (AI) castings. Eck is a leading production foundry for structural AI castings, serving multiple markets where outstanding performance and competitive costs are required. Eck is also a preeminent experimental research facility, involved in multiple research activities in high-performance AI alloys and production processes. PNNL has expertise in AI metallurgy, ultrasonic processing and techniques, and novel heat-treatment techniques. Therefore, this project leverages the respective strengths of PNNL and Eck to open new markets and opportunities for AI castings through an improvement in casting performance and reduction in costs.

The project proposes to use ultrasonic and heat-treatment techniques to lower the cost penalty inherent in the use of primary AI alloys and address mechanical properties issues in AI castings produced by secondary (i.e. recycled AI) alloys. Thus, the project objectives were to assess the effectiveness of (1) ultrasonic processing for modifying the deleterious intermetallic phases in recycled aluminum alloys, and (2) PNNL's proprietary heat-treatment technique for energy reduction and property improvement. The first objective was achieved by ultrasonic processing of primary and secondary AI alloy (A356) in molten state and characterizing the as-cast microstructure using optical and electron microscopy. The second objective was achieved by heat-treating AI alloy A206 using PNNL's proprietary heat-treatment technique.

The ultrasonic experiments on A356 alloy suggest ultrasonic processing can break the deleterious intermetallic phases into shorter segments. Additional research is needed to optimize processing parameters and validate the improvement in mechanical properties. If implemented commercially, ultrasonic processing can enable cheaper and high-performance Al castings made of recycled Al with the additional benefit of drastically reducing greenhouse gas emissions by reducing the need for primary Al. Heat-treatment experiments on A206 alloy suggest that PNNL's technique can significantly shorten the heat-treatment duration and lower the required temperature while delivering as good, or better, mechanical properties as standard heat-treatment practices. Additional research is needed to scale-up this technique to prototype castings. If implemented commercially, this technique can provide high-performance castings in shorter time and with reduced energy consumption.

Thus, overall, this project successfully accomplished its objectives and helped de-risk new technologies, and provided a pathway to move laboratory-scale research to industry adoption.

Summary of Research Results

Introduction

The U.S. transportation industry is the biggest user of aluminum (AI) castings. In 2015, 17.84 million light vehicles (cars and trucks) were sold [1]. A typical vehicle contains ~400 lbs AI (~180 Kg) (~73% as castings), which represents a number that is expected to rise as high as ~500 lbs (~230 Kg) in 2025 [2]. Considering that a typical full-size sedan weighs anywhere between 2000–3000 lbs (~900-1360 kg), greater use of low-cost and high-performance AI castings can contribute significantly to cost-effective vehicle weight reduction. However, cost and mechanical performance of AI castings are two key long-standing industry challenges that need to be overcome to enable greater lightweighting in mass-market vehicles. In other words, if the cost of AI castings could be reduced and their mechanical performance enhanced, even greater market penetration (e.g., by replacing ferrous components) is possible.

One way to lower the cost for Al castings is to use "secondary" Al alloys (i.e., those produced from recycled Al), due to their ~16% lower cost than the "primary" Al produced via electrolytic reduction of alumina. However, many secondary Al alloys allow up to 0.60% Fe (as compared to 0.1-0.2% Fe in primary Al alloy) leading to the formation of iron-intermetallic phases in the microstructure. These intermetallics are detrimental to the tensile properties and elongation of the castings as compared to the primary Al alloy. Thus, to keep costs low, Al may be skipped in favor of cheaper, but heavier, cast-iron. Further, irrespective of the choice of primary vs. secondary Al alloy, Al castings are often subjected to long duration heat treatments, such as solutionizing and aging, to improve their strength and ductility. Such heat treatments further add to the overall cost of the castings. Thus, techniques need to be developed that can help lower the cost of Al castings and enhance their performance (e.g., by enabling secondary Al alloys to overcome mechanical property limitations and by lowering the cost of heat-treatments).

Objectives

The goal of this project is to develop strategies to help lower the cost of AI castings by enabling secondary AI alloys to overcome the mechanical property limitations and by lowering the cost of heat-treatments. We propose to use two different processing techniques to achieve this goal. The first is an ultrasonic technique that enhances the mechanical properties by processing the molten secondary AI alloy, while the second uses a PNNL proprietary technique to shorten the solutionizing duration of the AI alloy. Thus, the project objectives are to:

- Develop process parameters for the ultrasonic technique leading to improvement in mechanical properties of the selected secondary AI alloy (i.e., containing "high" Fe %).
- Develop an alternate heat-treatment (with a shorter duration and/or lower temperature) leading to similar mechanical properties as the conventional heat treatment for the selected Al alloy.

Experimental Approach

<u>Materials</u>: Eck Industries has identified A356 AI and A206 AI as the alloys of interest for the purpose of ultrasonic process development and alternate heat-treatment development, respectively. The standardized chemistry of these alloys is listed in Table 1 [3] and both alloys were cast by Eck. Ingots of alloy A356 were cast with Fe < 0.1 wt.% and 0.9 wt.% to mimic primary (low Fe %) and secondary (high Fe %) version of this alloy, respectively. Accordingly, these two chemistries are referred to as "low Fe" and "high Fe", respectively, in this report.

Table 1: Standardized compositions of the A356 AI and A206 AI alloys.					
Alloy	Composition (wt.%)				
A356.0 AI	6.5% to 7.5% Si	0.25% to 0.45% Mg	0.2% Fe	0.2% Cu	
A206.0 AI	4.2% to 5.0% Cu	0.15% to 0.35% Mg	0.2% to 0.5% Mn	0.15% to 0.3% Ti	

<u>Ultrasonic Testing</u>: Figure 1a shows the bench-top setup developed for the ultrasonic experiments. Approximately 125 g of the A356 alloy were melted in the furnace at ~650°C in an alumina crucible. A 20 kHz Ti-6AI-4V ultrasonic probe was inserted into the furnace via an opening in the top and dipped into the molten metal. The ultrasonic impulses were applied in the melt by the probe and the key variable studied was the ultrasonic power (by adjusting the amplitude), keeping the treatment time and temperature constant at 2 min. and ~620°C, respectively. The ultrasonication temperature of 620°C is above the liquidus temperature (615°C) for A356 [3]. A limited number of ultrasonic experiments on high-Fe A356 were also conducted at ~615°C. The molten alloy, with or without ultrasonic processing, was poured into a graphite crucible at room-temperature and allowed to cool naturally, resulting in a "hockey-puck" shaped casting (Fig. 1b). Cast sample produced without ultrasonic processing are termed as "control samples" and compared against those produced by subjecting the molten alloy to ultrasonic pulses.



Figure 2: (a) As-cast dog-bone shaped A206 AI tensile bar; blue cylinders schematically show samples machined for heat-treatment experiments. (b) Standard solutionization heat-treatment temperature-time profile. T4 treatment involved natural aging the sample for 5 days after water quenching (E). T7 treatment involved an additional heating step (after T4) at 200 °C for 4 hours. Source: PNNL.

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The as-cast puck was sectioned longitudinally and samples were polished by traditional metallography techniques and analyzed by optical and scanning electron microscopy (SEM). Backscatter mode was used for SEM imaging and energy dispersive spectroscopy was used for composition analysis. ImageJ software [4] was then used to analyze the SEM images to quantify microstructural features such as the size and volume fraction of dendrites and intermetallics. The dendrite size (also referred to as "cell size" in the literature [5]) was determined according to the procedures described in [6]. Significant differences in the morphology of the intermetallic precipitates in the two variants of the A356 AI necessitated the use of different methods to quantify the intermetallic size in the respective alloys. Thus, in the low-Fe variant, the intermetallic size was defined as the diameter of the circle fitting to the average area of the precipitates while for high Fe, intermetallic size was defined as the longest dimension of the β needles. Figures 1c and 1d schematically show how the dendrite and intermetallic sizes were determined.

<u>Heat-treatment Testing</u>: The A206 Al alloy was permanent-mold cast by Eck Industries in the form of tensile bars seen in Figure 2(a) and test samples for heat-treatments were machined from these tensile bars. The samples for heat-treatments were machined to 15 mm long x 10 mm dia. sections (shown schematically as blue cylinders in Figure 2(a)) and to 101.6 mm long x 12.7 mm dia. bars. The shorter (15 mm) samples were used for hardness measurements after different heat-treatments. The longer (101.6 mm i.e. 4") bars were used for tensile testing performed at Eck Industries. The microstructures of heat-treated samples were characterized by SEM.

To establish a "baseline" mechanical properties, test samples were subjected to TheseT4 heattreatment. With reference to Fig. 2b, T4 comprises pre-treatment at ~510°C (A-B) with a 1.5 h ramp to the pre-treatment temperature, followed by solution treatment for 12 h at ~525°C (C-D), followed by water quenching (D-E) and finally, room temperature aging for 5 days (natural aging for T4 temper is generally defined as a minimum of 3 days). For some T4 samples, the solution heat-treatment was interrupted by water quenching at 2, 4, or 8 hours, followed by roomtemperature aging for 5 days. T4 heat-treated samples were further subjected to a T7 heattreatment at 200°C for 4 h. These various heat-treatments are termed as "standard" in the text implying they were performed in a conventional furnace. The heat-treated samples were characterized by hardness measurements (HRB: 1/16" Rockwell spherical indenter, 100 kg load) to evaluate the influence of heat-treatment on the mechanical properties. If there was a

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scheduling delay between T4 and T7, the hardness was also measured just before starting the T7 treatment.

As a comparison to the standard heat-treatment described above, test samples were subjected to the solutionization treatment by PNNL's proprietary electrical heating setup for various times and temperatures, followed by water quenching and subsequent T4 treatment for tensile testing or T7 treatment for hardness measurements. PNNL's electrical heating treatments were generally at lower temperatures and/or for shorter times relative to the standard treatment. Hardness was used as a metric to compare the efficacy of electrical heating approach to the standard heat-treatment. As the standard treatment involves pre-treatment and higher solutionization temperatures than those used in the electrical heating, some samples were solutionized in a furnace at similar times and temperatures as the electrical heating treatment for a direct comparison between the two heat-treatment approaches.

Results & Discussion

Ultrasonic Testing

<u>Low-Fe without and with u/sonics</u>: Figures 3a and 3b show backscattered electron images of the as-cast microstructure of the low-Fe A356 Al, without and with ultrasonication, respectively. The microstructure in general consists of α -Al dendrites, Al-Si eutectic, Mg₂Si and intermetallic compounds. Some intermetallics appear to be Chinese script-like phases referred to as α -phase in the literature. However, in our case (Fig. 3c) these phases seem to be the π -phase with an approximate composition of Al₈Mg₃FeSi₂ [7].

<u>High-Fe A356 without and with u/sonics</u>: Figures 4a and 4b show backscattered electron images of the as-cast microstructure of the high-Fe A356 Al, without and with ultrasonication, respectively. The microstructure in general consists of α -Al dendrites, Al-Si eutectic and needle-like Fe-containing intermetallic phase. Fig. 4c shows a higher magnification of the ultrasonicated sample and Fig. 4d shows its corresponding x-ray map. EDS analysis identified the needle-like intermetallics in the high-Fe samples as β -type Al₅FeSi precipitates. These precipitates are detrimental for ductility due to their high aspect ratio [6]. Figure 4e is a close-up of a region in the ultrasonicated high-Fe sample and shows numerous cracks in the Al₅FeSi phase, as indicated by the arrows. Such cracks were not observed in the control sample (i.e. cast without ultrasound). Therefore, it seems that ultrasonication was successful in cracking the brittle intermetallic phase and hence, lowering the aspect ratio. Such microstructural refinement



may help reduce the deleterious effect of such needle-like intermetallics on the mechanical properties of the casting [8].

<u>Comparison of low-Fe and high-Fe A356</u>: Comparing Figs. 3 and 4, the images show that the microstructures of high Fe samples are quite different than low Fe for both control and ultrasonicated samples. For example, in high Fe case, ultrasonication does not appear to change the dendrite or intermetallic sizes and the latter have a needle-like morphology, ~20 μ m



long. On the other hand, the low Fe sample has a smaller dendrite and intermetallic volume fraction and the intermetallics seem restricted to short lengths (~ 4 μ m) in inter-dendritic spaces. The precipitate morphology can also be affected by the cooling rate. However, since the experimental procedures for low and high Fe cases were kept the same, the cooling rate is expected to be the same and the presence of β -type precipitates is associated with the high Fe content.

Dendrite and intermetallic size trends with ultrasonic treatment: Figure 5 summarizes the dendrite size and intermetallic size in the high-Fe and low-Fe samples and under different casting conditions (without ultrasound, and with ultrasound at two different ultrasound power levels). The high-Fe samples show a slight increase in both dendrite and intermetallic sizes with the ultrasonication (Fig. 5a). Similar image analysis for low-Fe shows a slight increase, and then a decrease, in the dendrite size with higher ultrasonication powers. Intermetallic size on the other hand, slightly decreases with ultrasonication. It is important to note that some of these measurements are within the error included in image analysis. This leads to inconclusive results on the relationship between the ultrasonication and microstructure development, especially for the dendrite size. Under the test conditions employed in this work, the morphology of the intermetallics seems to be independent of the ultrasonication treatment in both low and high-Fe alloy.

Heat-treatment



<u>Hardness Measurements</u>: As part of the second objective of the project, hardness and microstructures of the A206 alloy are compared for the conventional and PNNL heat treatment procedures. Figure 6 summarizes the hardness values of samples that were solutionized for different durations (0, 2, 4, 8, or 12 hours) and were then naturally aged (5 days), followed by the T7 treatment. The data shows show that after solutionization + 5 days natural aging (T4 temper), the hardness increases to ~65 HRB. Subsequently, after the T7 temper treatment, the hardness increases to ~65-70 HRB range, with the hardness increasing with increasing duration of the prior solutionization step. This increase in hardness in A206 alloy upon T7 aging is associated with the precipitation hardening due to the formation of nano-scale Al₂Cu precipitates [3]. A longer solutionization treatment (e.g. 12 hours) results in greater amount of Al₂Cu (in the as-cast state) to dissolve and put Cu in solution. A higher Cu% in solution results in a higher volume fraction of fine Al₂Cu precipitates, and hence, greater precipitation hardening, during subsequent T7 heat-treatment step.

Table 2 compares three "standard" heat-treatment experiments with PNNL heat-treatment procedures. The phrase "standard" refers to the conventional heat-treatment practice (refer to Fig. 2b) employed by Eck Industries and heat-treatment industry in general [3]. For example, in the heat-treatment labeled Standard 8 h, the "8 h" refers to the 8 hours duration of solutionization step at the highest temperature. It is noted that although (in this example), the solutionization is performed for 8 hours, the overall heat-treatment process is actually much

Table 2: Comparison of PNNL and standard heat-treatments and the resulting hardness. The "Hardness (Solutionized)" column values were measured before the T4 temper (5 days natural aging) step.

Heat Treatment	Ramp Time (h)	Soak Time (h) and Temperature (°C)	Total Process Time (h)	Hardness (Solutionized) HRB	Hardness (T7 Aged) HRB
Standard "0 h"	1.5	2 h at 517°C	3.5	23.2 ± 1.5	63.7 ± 0.7
Standard "8 h"	1.5	2 h at 510°C + 8 h at 522°C	11.5	28.3 ± 0.4	68.1 ± 0.7
Standard "12 h"	1.5	2 h at 510°C + 12 h at 522°C	15.5	22.0 ± 3.6	70.8 ± 1.0
PNNL "2 h"	0.1	2 h at 500°C	2.1	29.1 ± 3.0	68.0 ± 0.6

longer (11.5 hours in this case) when the ramp-up and soak times are also added. The PNNL 2h heat-treatment refers to the experiments where the solutionization was performed by PNNL's proprietary electrical heating setup for 2 hours. This was followed by standard T7 tempering heat-treatment. PNNL's approach had a shorter ramp up time and does not have a pre-treatment step (step A-B in Fig. 2b). The data in Table 2 shows that when compared to the standard, PNNL's heat-treatment at 500°C 2 h yields similar or higher hardness in both, assolutionized and in T7 tempers. Further, PNNL 2 h method's hardness values are identical to the standard at 8 h (Total processing time = 11.5 h), indicating that PNNL's method is about 6 times more efficient than the standard in terms of the processing time.

Figure 7 summarizes the hardness results obtained for heat-treatments with solutionization temperatures lower than that used in conventional practice (i.e. 525°C). The data in Figure 7 suggests that furnace solutionization treatments at 450°C and 475°C failed to solutionize the ascast A206 AI as both, solutionized and T7 hardness, are far lower than the as-cast hardness



(54.5 ± 1.2 HRB). Further, since the solutionized and T7 temper hardness are almost similar, this indicates that that there was minimal Cu dissolved in the matrix during solutionization to cause any appreciable hardening during the aging step. In turn, minimal concentration of Cu dissolved in the matrix is attributed to the slow kinetics of dissolution of Al₂Cu particles during solutionization (at 450°C and 475°C) and that these solutionizing temperatures were considerably lower than the equilibrium solvus temperature (~525°C, for an Al-Cu alloy containing ~ 4.6 wt% Cu). Contrary to the hardness results of standard heat-treatments, Figure 7 also shows that the PNNL heat-treatments at the same temperatures (at 450°C and 475°C) and for the same total processing time result in significantly higher hardness than furnace treatments, both in the as-solutionized and T7 tempers. In PNNL heat-treatments cases, the hardness after aging is much greater than in the as-solutionized state. In other words, even though the PNNL heat-treatment solutionization was performed below the solvus temperature, such heat-treatment was still able to dissolve Al₂Cu precipitates and re-precipitate fine Al₂Cu during the aging.

<u>Microstructural Analysis:</u> Microstructural analysis of the T7 heat-treated samples in Figure 8 also confirms the efficiency of PNNL's method, relative to standard, in solutionizing the large Al₂Cu precipitates that are present in the starting as-cast microstructure. Backscattered SEM images in Figure 8 show that while none of the heat-treatments completely solutionized the Al₂Cu precipitates, the undissolved volume fraction of Al₂Cu (the bright phase) is comparable. The major difference in these images is that while the standard treatments appear to be more successful in dissolving precipitates along the grain boundaries, the PNNL modified treatment seems to result in "cleaner" grain interiors suggesting it is better at dissolving precipitates in the



Figure 8: Backscattered SEM microstructures of A206 alloys solutionized and aged by: (a) standard "0 h"; (b) standard "12 h"; and (c) PNNL- 2 h. Al₂Cu volume fractions calculated form these images are: 0.5, 0.6 and 0.9 %, respectively. Source: PNNL.

grain interior. Additional microstructural analysis is needed to clarify the effects of different heat-treatment approaches.

Table 3 summarizes the tensile properties of A206 test bars (4" long) that were heat-treated according to the "PNNL 2h" treatment shown in Table 2, followed by T4 tempering. These properties are also compared against Eck Industry's tensile data on A206 AI that has also been heat-treated to the T4 temper following their conventional heat-treatment practice. The data in Table 3 shows that tensile properties of A206 AI, produced by PNNL's heat-treatment with only 2 hour solutionization treatment, are at par or somewhat better than the properties in A206 AI heat-treated (12 hours solutionization treatment) by conventional practice.

Table 3: Comparison of tensile properties of A206 AI heat-treated by PNNL method (500°C for 2
hours followed by T4 tempering) vs. conventional practice at Eck.	

Property	Average of 6 samples	Average excluding 1 premature failure	Typical Commercial values -Eck
Tensile strength	359 ± 40	375 ± 12	345
MPa (ksi)	(52 ± 6)	(54 ± 2)	(50)
Yield Strength	242 ± 10	241 ± 10	205
MPa (ksi)	(35 ± 1)	(35 ± 1)	(30)
Total Elongation (%)	11 ± 6	14 ± 4	10

Conclusions

This project was performed in collaboration with Eck Industries and investigated two techniques with the overall goal to lower the cost of Al castings. The first technique applied ultrasonic pulses to the molten alloy A356, with low- and a high-Fe compositions, with the expectation that ultrasonic processing could refine the high-aspect ratio β -Al₅FeSi intermetallic precipitates that could enhance the ductility of the cast alloy. The second technique was a PNNL proprietary heat treatment technique with the expectation to shorten the solutionization duration (and/or lower the solutionization temperature) of A206 Al. Based on the results of this work, the following conclusions can be drawn:

- 1) Ultrasonic treatment in low-Fe A356 Al alloy resulted in slight refinement in sizes of dendrites and intermetallic phases.
- Ultrasonic treatment in high-Fe A356 AI resulted in a slight increase in the sizes of dendrites and β-AI₅FeSi intermetallics. Formation of these precipitates was due to the high Fe content

and/or possibly slow cooling rate during casting such that ultrasonic treatment under the conditions used in this work was not enough to transform these precipitates.

- 3) Ultrasonic treatment in high-Fe A356 AI around the liquidus temperature showed evidence of fracture in the β-AI₅FeSi intermetallics. Additional work is needed to optimize the processing conditions to refine the intermetallics size and to determine the effect of such refinement on mechanical properties.
- 4) When compared to the standard heat-treatment practice of A206 AI, PNNL's heat-treatment method resulted in similar hardness and microstructure, and similar or somewhat better tensile properties (yield strength, ultimate tensile strength and ductility). However, PNNL's method required ~6-times shorter solutionizing time and ~25°C lower solution temperature relative to the standard practice.

Subject Inventions

1. PNNL Invention disclosure (IPID 32446-E): A method for heat-treatment of Al alloys.

Publications and Presentations

- 1) DOE-VTO FY 2019 Annual Progress Report.
- 2) DOE-VTO FY 2020 Annual Progress Report.
- DOE-VTO Annual Merit Review presentation, Project ID# mat158, 2019, Washington, D.C. USA.
- DOE-VTO Annual Merit Review presentation, Project ID# mat158, 2020, Washington, D.C. USA.

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