

A Methodology to Predict the Effects of Quench Rates on Mechanical Properties of Cast Aluminum Alloys

by

Shuhui Ma

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Richard D. Sisson, Jr., George F. Fuller Professor

Director of Manufacturing and Materials Engineering

Abstract

The physical properties of a polymer quench bath directly affect the cooling rate of a quenched part. These properties include the type of quenchant, concentration, and agitation level. These parameters must be controlled to optimize the quenching process in terms of alloy microstructure, properties, and performance. Such data is scarce for cast aluminum alloys in the literature and a quantitative measurement of the effects from individual process parameter is not available. In this study statistically designed experiments have been performed to investigate the effects of the process parameters (i.e. polymer concentration and agitation) on the quenching behavior of cast aluminum alloy A356 in aqueous solution of Aqua-Quench 260 using the CHTE quenching-agitation system. The experiments were designed using the Taguchi technique and the experimental results were analyzed using the Analysis of Variance (ANOVA) in terms of the average cooling rate. It is found that the average cooling rate dramatically decreases with the increase in polymer concentration. The agitation only enhances the average cooling rate at low and medium levels. Based on the results from ANOVA, the process parameter that affects the average cooling rate most is the polymer concentration, its percentage of contribution is 97%. The effects from agitation and the interaction between polymer concentration and tank agitation are insignificant.

The mechanical properties of age-hardenable Al-Si-Mg alloys depend on the rate at which the alloy is cooled after the solutionizing heat treatment. A model based on the transformation kinetics is needed for the design engineer to quantify the effects of quenching rates on the as-aged properties. Quench Factor analysis, developed by Staley, is able to describe the relationship between the cooling rate and the mechanical properties of age-hardenable aluminum alloys. This method has been previously used to successfully predict yield strength and hardness of wrought aluminum alloys. However, the Quench Factor data for aluminum castings is still rare in the literature. In this study, the Jominy End Quench method was used to experimentally collect the time-temperature and Meyer hardness data as the inputs for Quench Factor modeling. Multiple linear regression analysis was performed on the experimental data to estimate the kinetic parameters during quenching. Time-Temperature-Property curves of cast aluminum alloy A356 were generated using the estimated kinetic parameters. Experimental verification was performed on a L5 lost foam cast engine head. The predicted hardness agreed well with that experimentally measured.

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Shuhui Ma

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Chapter I. Introduction

Cast Al-Si-Mg alloys have been widely used in automotive and aircraft industries for their good properties and high strength-to-weight ratio [1-3]. Intensive studies of this cast aluminum family has been found in the literature in terms of enhancing the mechanical properties [2, 4-8]. It is well known that the heat treatment is one of the important methods for improving the mechanical properties of aluminum alloys [5]. The heat treatment of age-hardenable aluminum alloys involves solutionizing the alloys, quenching, and then either aging at room temperature (natural aging) or at an elevated temperature (artificial aging) [3, 9].

Polymer Quench of Aluminum Alloys

Quenching is a crucial step to suppress the precipitation to retain the supersaturation of solid solution, control the distortion, and minimize the residual stress in aluminum alloys. Quenching media commonly used for aluminum alloys include brine solution, water, and polymer solutions [10-12]. The physical properties of polymer quench bath directly affect the cooling rate of a quenched part. These properties include the type of quenchant, its temperature, concentration, and agitation level [10, 13, 14]. These parameters must be controlled to optimize the quenching process in terms of alloy microstructure, properties, and performance. Polymer quenchants are advantageous because they can be disposed of safer and easier than oils but still maintain similar quenching performance [15-18]. They are flexible in

quenching because their concentration in water can be varied to obtain the desired cooling rates. Also, polymers reduce the risk of fire and make it easier to control the cracking and distortion that water can often cause [18-20]. Cost is also an advantage to using polymers to thicken water over using oils. Intensive investigation has been carried out by many researchers to study the effect of water temperature, the concentration of polymer solution on the mechanical properties of wrought aluminum alloys [21-23]. However, such data is scarce in the literature for cast aluminum alloys. Therefore, in this study the Taguchi technique is employed to design the test matrix for estimating the effects of agitation level and polymer concentration on the cooling rate, mechanical properties, and aging kinetics of cast aluminum alloy A356 in a PAG-based polymer quenchant. The experimental result is analyzed using analysis of variance (ANOVA).

Quench Factor analysis for property prediction

The mechanical properties of age-hardenable Al-Si-Mg alloys, to a large extent, depend on the rate at which the alloy is cooled after the solutionizing treatment. A model based on the transformation kinetics is needed for the design engineer to quantify the effect from the quenching process. Quench Factor analysis, developed by Staley, is able to describe the relationship between cooling rate and the mechanical properties of age-hardenable aluminum alloys. This analysis assumes the precipitation of secondary phase during continuous cooling is additive and can be described by the nucleation

and growth kinetics [9, 24-27]. This method has been previously used to successfully predict yield strength and hardness of wrought aluminum alloys [26, 28-34]. However, the application of Quench Factor analysis for aluminum castings is still rare in the literature. In this study, the Jominy End Quench method [35] is used to collect experimental data needed for Quench Factor modeling. Numerical analysis is performed on the experimental data to estimate the kinetic parameters of cast aluminum alloy A356. Time-Temperature-Property curves are generated with the kinetic parameters. Based on this, the mechanical properties of cast aluminum alloy A356 can be predicted using the Quench Factor models.

Research objective

The objective of this research is to develop a methodology to establish the relationships between the process parameters, structure and properties for heat-treated cast aluminum alloy A356. Special emphasis is focused on characterization of the quenching behavior of cast aluminum alloy A356 in polymer solution for different process parameters and the estimation of the Quench Factor parameters based on the precipitation kinetics of cast aluminum alloy A356, which can be employed for property prediction.

Thesis organization

The thesis is divided into four chapters. Chapter I is an introduction that gives an overview of this study, the objectives and the thesis organization. Chapter II is a thorough review of the relevant literature. The literature review includes the investigation of various heat treatment methods for cast Al-Si-Mg alloys, specifically cast aluminum alloy A356, in terms of enhancing the mechanical properties and the history of Quench Factor analysis development for property prediction with the known thermal history and precipitation kinetics. Chapter III is a series of two papers that emphasize two different aspects of this research. Paper #1, titled “The Effects of Polymer Concentration and Agitation on the Quench Performance of Polymer Quenchant Aqua-Quench 260” by *Shuhui Ma, Md. Maniruzzaman, and Richard D. Sisson, Jr.*, describes the effect of process parameters, polymer concentration and agitation, on the quenching characteristics of cast aluminum alloy A356 using the Taguchi technique and the Analysis of Variance (ANOVA). Paper #2, titled “A Methodology to Predict the Effects of Quench Rates on Mechanical Properties of Cast Aluminum Alloys” by *Shuhui Ma, Md. Maniruzzaman, D.S. MacKenzie, and Richard D. Sisson, Jr.*, describes a procedure for estimating the kinetic parameters needed for the Quench Factor models and the experimental verification with a lost foam cast A356 engine head. Chapter IV provides the conclusions based on this research.

Chapter II

Heat Treatment of Cast Al-Si-Mg

Alloys - A Literature Review

1.0 Heat treatment of cast Al-Si-Mg alloys

Cast Al-Si-Mg alloys have been widely used in automotive and aircraft industries for their good properties and high strength-to-weight ratio [2, 3]. The castings are usually heat-treated to obtain the desired combination of strength and ductility. The desired mechanical properties for these applications include high yield/tensile strength, good fracture toughness, and excellent resistance to fatigue. The heat treatment of cast Al-Si-Mg alloys is usually investigated from the following three aspects: solutionizing, quenching, and aging.

Typical heat treatment process for cast aluminum alloy A356 is T6 condition, which consists of a solution heat treatment, quenching and aging at an elevated temperature. ASTM Standard B917-01 designates 6-12 hours at 540°C, hot water quench, and then 2-5 hours at 155°C for sand-cast A356 [36], while permanent mould cast bars require 4-12 hours solutionizing at 540°C and 2-5 hours aging at 155°C [36]. AFS suggests the T6 heat treatment for A356 is to solutionize at 538°C for 12 hours followed by 3-5 hours artificial aging at 155°C for sand casting and 227°C for permanent mold castings [37]. However, variations of a standard T6 heat treatment were investigated by researchers for Sr-modified and unmodified cast aluminum alloy A356 in terms of the effects on the mechanical properties.

1.1 Effect of Solutionizing temperature and time

A solutionizing treatment of cast Al-Si-Mg alloys in the range of 400-560°C dissolves the hardening agents (Mg_2Si particles) into the α -Al matrix, reduces the micro-segregation of magnesium, copper, manganese, and other addition elements in aluminum dendrites, and spheroidizes the eutectic silicon particles to improve the ductility [3, 8]. The amount and rate of dissolution increase with increasing solution treatment temperature, but the temperature is limited by the solidus temperature.

The desired solutionizing treatment time and temperature, to a great extent, depend on the casting method, the extent of modification, and desired level of spheroidisation and coarsening of silicon particles. Work has been done in the past to study the effects of both solutionizing time and temperature on the mechanical properties of cast aluminum alloy A356.

Kelly et al investigated the effects of variations from T6 standard treatment on the hardness, ductility, and UTS of aluminum alloy A356 cast in a permanent mold with and without strontium modification [4]. The main variables considered in the experiments were solutionizing time and temperature. The as-cast samples were solutionized for various times ($t=2, 4, 8, 16$ and 32 hours) at $520^\circ C/540^\circ C$ and aged at $160^\circ C$ for 6.5 hours [4]. The highest hardness was obtained at a short solutionizing time (2 hours) for both

unmodified and modified A356, while the highest ductility wasn't achieved until the samples were solutionized for 8 hours at the same temperature, as shown in Figure 1 [4]. A slight change in solutionizing temperature didn't cause much variation in hardness, ductility and UTS. It could also be seen from Figure 1 that the strontium modified samples exhibited higher elongation than the unmodified ones under all the heat treatment conditions reported in this study [4].

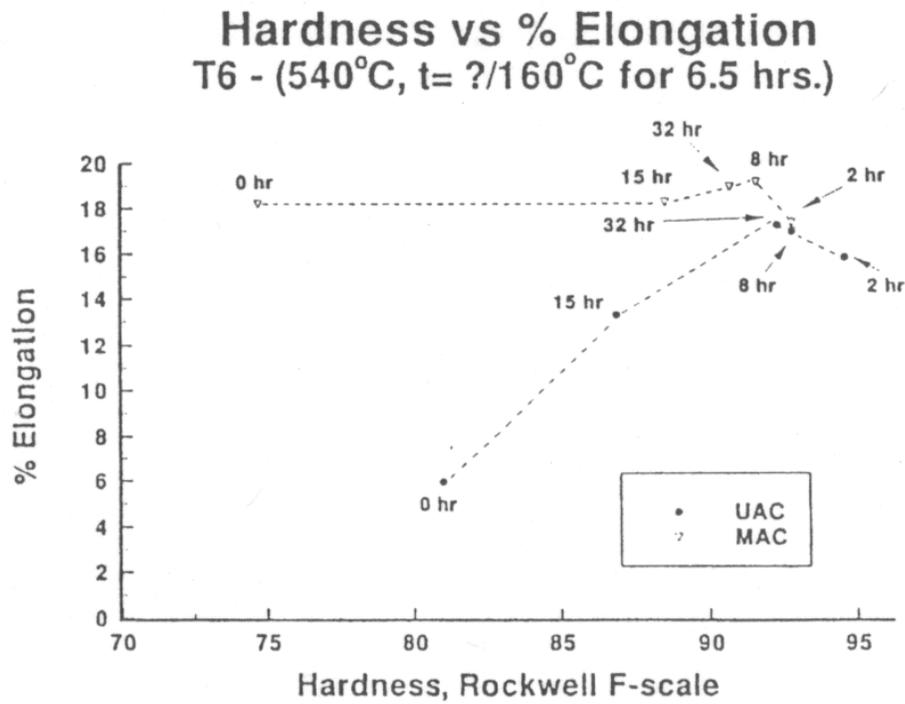


Figure 1. Hardness vs. elongation with respect to the variation in solutionizing time [4]

Mechanical properties of Al-Si-Mg alloys are related to the morphology of silicon particles (size, shape and distribution), aluminum grain size and shape, and dendritic parameters [38, 39]. Three factors, solidification rate,

modification, and heat treatment, can alter the silicon morphology from coarse and large needles into a fine and well-rounded form, thus improve the ductility of an alloy [5].

The influence of solidification rate, Strontium and Antimony addition, heat treatment, and their relationship with microstructure and mechanical properties of A356.2 alloy was studied by *Shabestari et al* [5]. Tensile test specimens, machined for both sand and permanent mold casting conditions, were solutionized at 540°C for 6 hours, quenched in 60°C water, and aged at 155°C for 4 hours. As-cast and heat-treated samples were examined. From the SEM micrographs, it was observed that a faster solidification rate in the thin wall castings or permanent mold castings promoted a faster nucleation and growth rate and resulted in a finer microstructure than sand cast condition, which was associated with higher mechanical properties [5]. The typical secondary dendrite arm spacing (SDAS) for aluminum A356 cast in a metal mold is in the range of 30-50µm and the size of silicon particles after solutionizing is between 2 and 20 µm [40]. In this study the average diameter of silicon particles was observed to decrease from 8.92µm to 7.74 µm after heat treatment for unmodified samples with 3mm thickness, while a slight increase of 1.12 µm to 1.54 µm was observed for Sr modified samples [5]. In terms of mechanical properties, the elongation, UTS, and yield strength of test bars decreased with increasing DAS for all the samples and increased

with the addition of Sr and Antimony modifiers [5]. And it was found that the tensile properties, best predicted from particles size and density, were most effectively improved by heat treatment than by solidification rate and modification [5].

A quantitative evaluation of the evolution of silicon particles during solution heat treatment was carried out by *H.M.Tensi* for sand cast aluminum A357 solidified at different rates [6]. The volume of eutectic silicon phase increased with the increase in solutionizing time for the range of 0.5 to 50 hours. The coarsening of silicon particles was observed to occur after the sample was solutionized at 540°C for 4 hours [6]. In order to investigate the kinetics of silicon growth and coarsening, two theories were presented. In these two theories, the process was described either as a purely diffusion-controlled silicon growth or mainly the coarsening of silicon phase by “Ostwald-Reifung” mechanism during the solutionizing treatment [6]. The equivalent diameter of silicon particles was plotted as a function of $(t)^{1/2}$ and $(t)^{1/3}$. Both plots showed a linear relationship of the equivalent diameter of silicon phase with $(t)^{1/2}$ or $(t)^{1/3}$. However, the “ \sqrt{t} law” showed a better relationship for a coarser microstructure which resulted from slow solidification rate [6]. 60% increase in the hardness was found by heat treatment process.

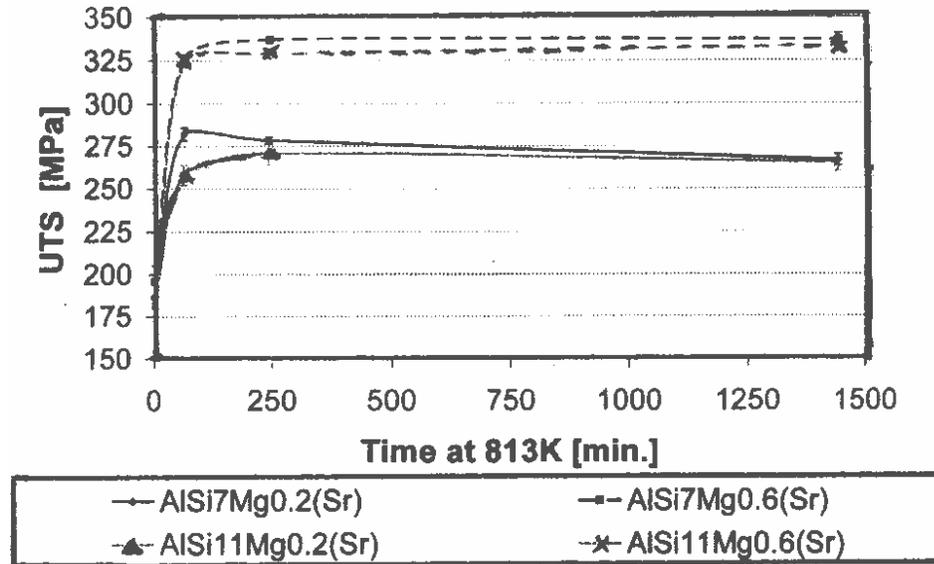


Figure 2. Ultimate tensile strength of modified alloys that have been solution heat treated at 813K, quenched in water and aged at 423K for 4 hours [7]

The effects of Mg and Si concentration in cast Al-Si-Mg foundry alloys was studied by *L. Pedersen et al* by comparing the mechanical properties before and after heat treatment [7]. The experiments were based on four main alloy compositions: Al-7Si-0.2Mg, Al-7Si-0.6Mg, Al-11Si-0.2Mg, Al-11Si-0.6Mg. The round tensile bars were solution heat treated in an air circulation furnace at 540°C for 1, 4 and 24 hours, immediately quenched in water at room temperature, and then artificially aged in an oil bath at 150°C for 4 hours [7].

The maximum strength (UTS) was obtained for all the compositions after 60 minutes of solutionizing and a prolonged solution heat treatment didn't lead to an increased strength, as could be seen from Figure 2 [7]. The strength was

mostly influenced by the Mg concentration in the alloy and was nearly independent of the silicon level. The alloy with higher Mg concentration showed higher strength, which was due to the formation of a higher density of hardening β' -Mg₂Si precipitates [7]. The combination of Mg and Si concentration was observed to affect the ductility. Higher silicon level led to a reduced ductility even after a long solution treatment, which resulted from the increased amount of Al-Si eutectic. In summary, solution heat treatment of the foundry alloys leads to two more-or-less competing changes in the microstructure [7]. On the one hand, microstresses from the formation of metastable β' -Mg₂Si precipitates lead to an overall reduction in ductility in the aluminum; on the other hand, the solution heat treatment leads to changes in the silicon's morphology, hence increases the ductility [7].

There were also findings from other researchers in the literature. *Rometsch et al* [41] showed that AC603 (Australian version of A356) alloy with a DAS of 50 μ m, a time of only 35 minutes was sufficient for complete dissolution of Mg₂Si and homogenization of Mg. Only coarser microstructures required longer solutionizing time. However, it had not yet been determined how the dissolution of Fe-containing phase would affect the mechanical properties of this alloy if the solutionizing time was reduced from 8 hours. *Taylor et al* [42] concluded that for castings having short solidification times (i.e. fine microstructures) the tensile strength and ductility were not adversely

affected by reducing the solutionizing treatment time from 8 hours to a few hours since the spheroidisation of Si particles occurred rapidly in the few hours at 540°C and continued more slowly thereafter. From this study it is safe to say the potential for savings of at least a few hours in process time appears to be significant. *Davidson et al* [8] tested the specimens machined from three different sources and found out reducing the solutionizing time of cast A356 from 8 to 4 hours had no effect on its fatigue endurance properties. *D. Emadi* [3] found in his study that the solutionizing time of 4 hours at 540°C gave optimal properties and reproducibility when coupled with cold/warm water quench, 6-12 hours pre-aging, and 6 hour aging at 155°C. Based on the above findings, it is possible to reduce the solutionizing time from 8 hours without significantly affecting the mechanical properties of cast aluminum alloy A356 although it is unrealistic to shorten it to below 1 hour.

Other than the standard T6 heat treatment, T5 without solutionizing step was also employed for heat treating aluminum castings. *P. Cavaliere et al* [2] studied the influence of both T5 and T6 heat treatments on the mechanical properties of thixocast aluminum alloy A356. The dimension of the specimens used in this study was 200mm in length and 18mm in diameter. The specimens were solutionized at 540°C for 1,2,4,8 and 16 hours, quenched, and aged at 160°C and 200°C for T6 condition, while other specimens were aged at the same temperature without solution treatment for T5 condition

[2]. The heat treatment effects were characterized by hardness, electrical conductivity measurements, and tensile tests. Different microstructural phenomena were observed to take place during the T6 heat treatment at high temperature. For short solutionizing times, the dissolution of intermetallic particles in the matrix resulted in the hardening of the alloy, while for longer solutionizing times, the spheroidisation of silicon particles led to the softening of the alloy [2]. The hardness reached the maximum at 4 hours solutionizing. The aging treatment both in T5 and T6 conditions produced an increase in mechanical properties. The aging temperature was observed to affect the ductility to a large extent, but didn't vary YS and UTS much [2].

Although 540°C is a recommended temperature for solutionizing cast aluminum alloy A356 by many organizations, other temperatures have also been successfully employed by some investigators. The activation energy of the coarsening process was calculated to be 80 cal/mole [43]. Hence, a small variation in the temperature can dramatically change the duration of the solutionizing time, e.g. the 12 hours at 530°C, necessary to achieve 18% elongation, can be done in two hours at 540°C and even 1/2-1 hour at 550°C [44]. Even though the increase in the solutionizing temperature can significantly reduce the time, localized melting of Fe-and Cu-containing particles at the grain boundaries needs to be aware of since it can reduce mechanical properties to some extent.

1.2 Effect of quenching rate

The objectives of quenching are to suppress the precipitation during quenching and to retain solute atoms and quenched-in vacancies in solution [45]. The best combination of strength and ductility is achieved from a rapid quenching. Cooling rates should be selected to obtain the desired microstructures and to reduce the duration time over certain critical temperature range during quenching, in the regions where diffusion of smaller atoms can lead to precipitation at potential defects [3].

The quenchants used for quenching aluminum alloys include water, brine solution and polymer solution [10-12]. Water used to be the dominant quenchant for aluminum alloys, but water quenching most often causes the distortion, cracking, and residual stress problems [10, 11, 19, 20]. Traditionally there are two ways to tackle these problems; one method is to increase the water temperature so that the temperature gradient between water and the part being quenched can be reduced [10]. It is reported that the water temperature affects properties of cast aluminum alloy A356 subjected to T6 heat treatment once the water temperature exceeds 60-70°C, with UTS and YS being significantly more sensitive than ductility [3]. However, the distortion problem can't be effectively solved by this method. The other method is to use the polymer solution. Quenching in polymer solution is used more widely nowadays since the distortion problem can be

effectively reduced by varying the polymer concentration and more uniform quench can be readily obtained [11, 13, 15-20].

Although a high quench rate is essential to achieve the high strength, in many cases, such a quench rate can't be used due to problems of high internal stress and distortion. This is especially true for cast components with the complex shapes and thin sections. To ensure that the minimum required strength is obtained throughout a cast component, the effects of quench rate on the strength of casting alloys need to be understood.

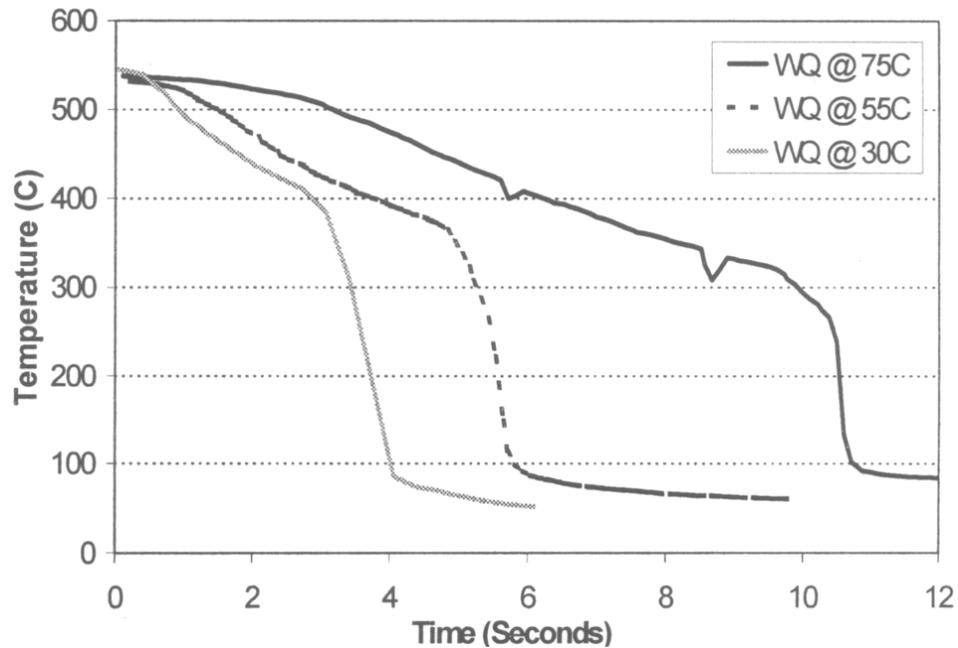


Figure 3. Effects of quench media temperature on cooling rates [3]

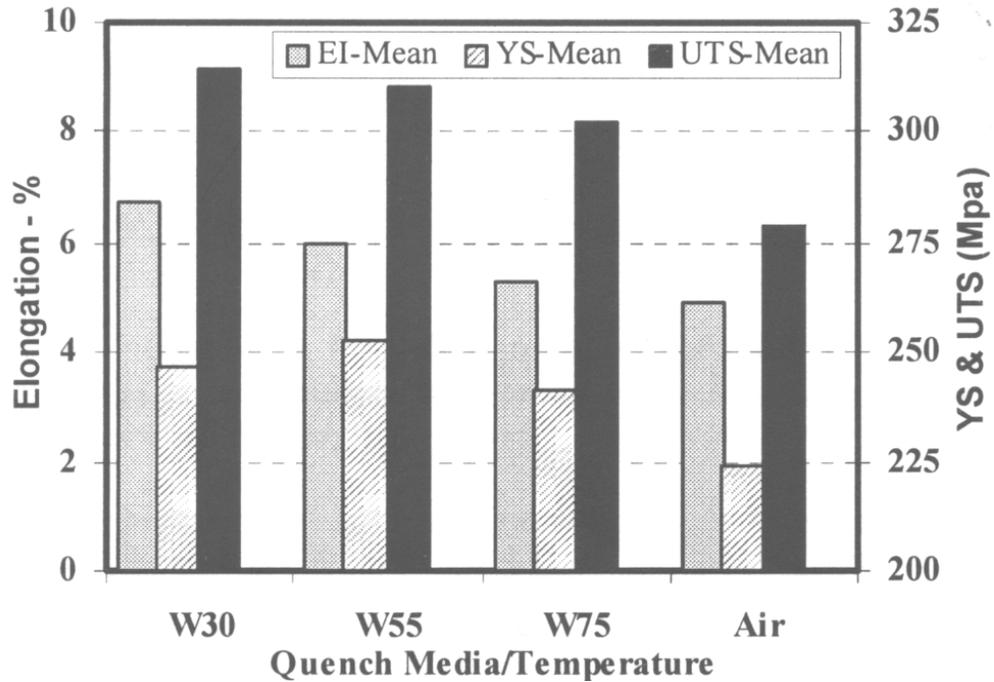


Figure 4. Effects of quench media on mechanical properties (4 hours solutionizing@540°C, quench, and 6 hours aging @170°C) [3]

Work was undertaken to address the quench sensitivity of cast Al-Si-Mg based alloys. The effect of water temperature on cooling rate of cast aluminum alloy A356.2 was investigated by *D. Emadi et al* [3]. Water at different temperatures, 30°C, 55°C, and 75°C, was used as quenchant in this study and air quench was performed for comparison purpose. It was found that increasing the water temperature or using air reduced the cooling rate and increased the chance of precipitation during quenching [3]. The cooling curves in Figure 3 showed the collapse of the steam blanket around the bars not only occurred in the critical temperature range of 290°C to 400 °C, but also extended to a lower temperature for the warm water quench, which explained the low cooling rate resulted from water quench at elevated

temperature. An examination of the effects of quench media on properties in Figure 4 showed that higher UTS, YS, and elongation were obtained from cold water quench although certain amount of warping could be resulted [3].

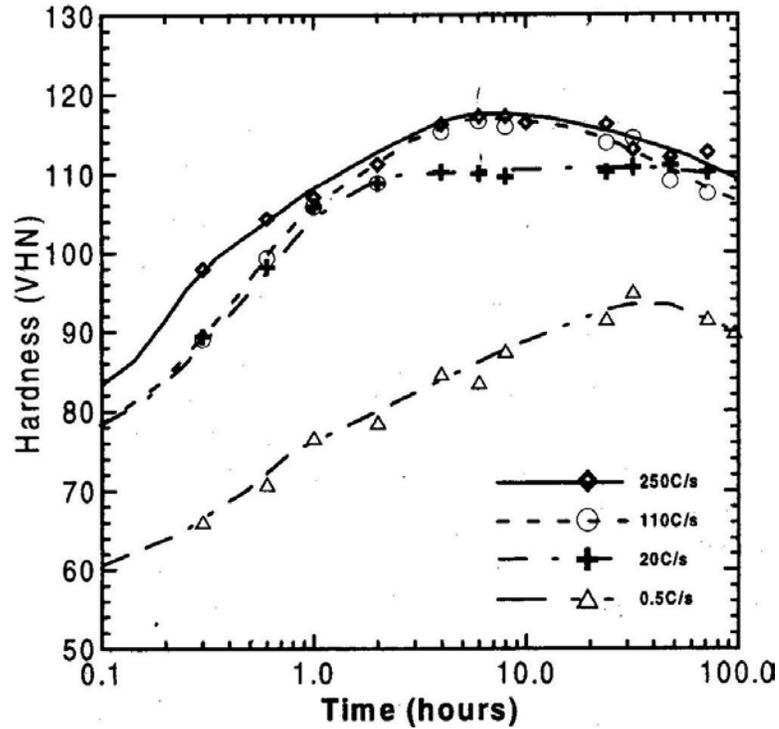


Figure 5. Hardness profile of cast aluminum alloy A356 vs. aging time at 170°C for different quench rates [45]

D.L.Zhang and L.Zheng [45] reported in their study of cast Al-7%Si-0.4% Mg alloy that the average quench rate within the temperature range of 200°C to 450°C was the most critical in influencing the strength [45]. Quench rates in the range of 0.5°C/sec to 250°C/sec were investigated after the samples were solutionized at 540°C for 14 hours and subsequently aged at 170°C for 6 hours. It was found the peak hardness wasn't affected by the quench rate when the quench rate was higher than 110°C/ sec. However, when the quench

rate was reduced to 0.5°C/ sec, the peak hardness decreased to only about 78% of the peak hardness obtained with 250°C/ sec, as given in Figure 5 [45].

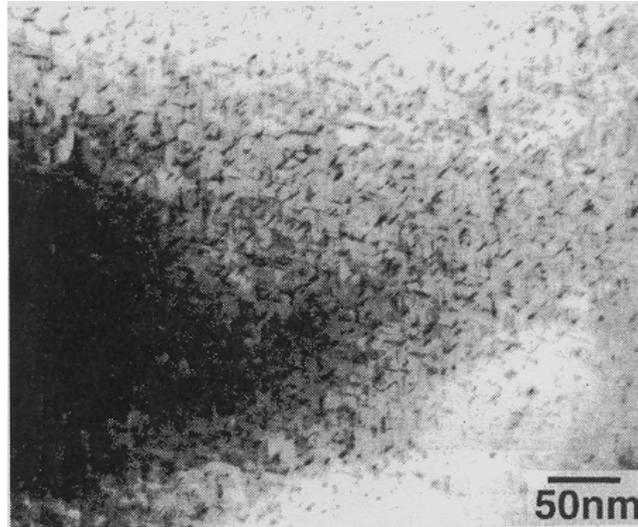


Figure 6. TEM micrograph showing β'' -Mg₂Si precipitates in the α -Al matrix of peak-aged A356 alloy corresponding to an average quench rate of 250°C/s. Beam direction: $\langle 112 \rangle_{Al}$ [45]

From the microstructure analysis, it was observed the size and shape of eutectic silicon weren't influenced by the quenching condition and the quenching condition only influenced the nature of Mg₂Si-type precipitates in the α -matrix from the subsequent artificial aging [45]. TEM examination of the peak-aged samples in Figure 6 showed only β'' - Mg₂Si precipitates (3-4nm diameter, 10-20nm length) were present in the matrix if samples were quenched at 250°C/sec. The number of precipitates decreased and the size increased slightly when the quench rate was lowered to 110°C/ sec [45]. However, for air quench (0.5°C/ sec), besides a high density of fine β'' - Mg₂Si precipitates (2-3nm diameter, 40nm length), a large number of areas that

contained coarse rods of β' - Mg_2Si precipitates (15nm diameter, 300nm length) and surrounding precipitate free zones (PFZs) were seen in the α -Al matrix, as shown in Figure 7 [45]. The yield strength and UTS of the peak-aged cast aluminum alloy A356 decreased respectively by 33% and 27% as the quench rate decreased from 250°C/sec to 0.5°C/sec [45].

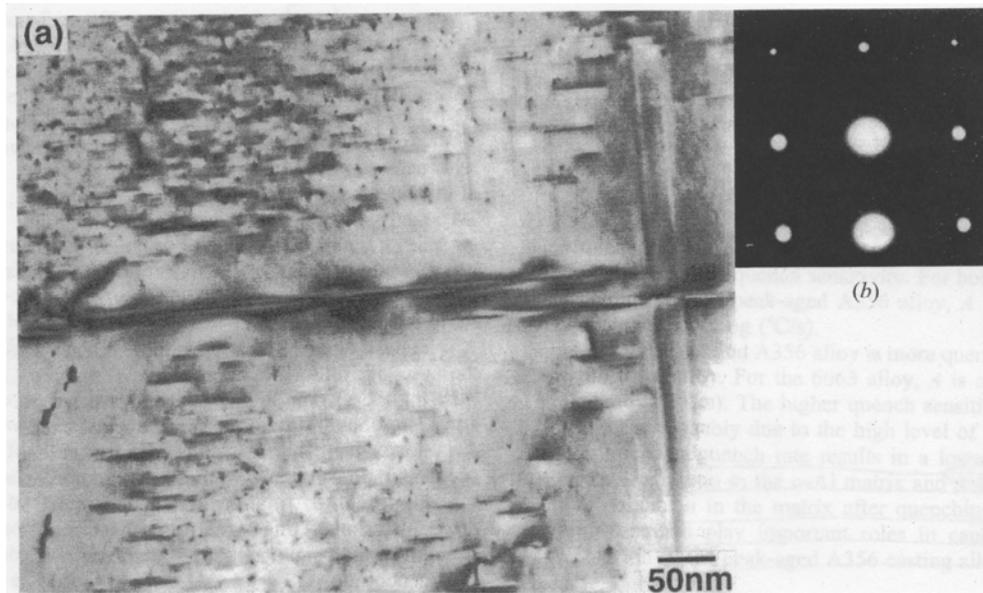


Figure 7. (a) TEM micrograph showing fine β'' - Mg_2Si and coarse β' - Mg_2Si precipitates and precipitate-free zones in the α -Al matrix of peak-aged A356 alloy corresponding to an average quench rate of 0.5°C/s; and (b) a selected area electron diffraction pattern of $\langle 112 \rangle_{Al}$ zone axis corresponding to (a) [45]

A reduction in the quenching rate was observed to lead to a reduced strength and an increased ductility in alloys with a high magnesium concentration by *L. Pedersen et al*, while the ductility of low magnesium alloys was relatively unaffected by a reduction in the quenching rate [7]. The reduction in strength was related to the lower density of hardening precipitates Mg_2Si formed,

which can be related to the amount of vacancies present. A reduced quenching rate allowed vacancies to move, partly cluster within the α -Al, and partly “disappear” out of the α -Al by diffusion to surfaces and probably also to areas near the silicon particles [7]. An increase in ductility with decreasing cooling rate for high-Mg alloy was attributed to the lower level of excess silicon in the matrix due to the formation of Mg_2Si . For high-Mg alloys, slow quench resulted in the formation of silicon precipitates at smaller size during the quenching. While for rapid quenching the entire growth of the Si precipitates took place during the subsequent aging and the relatively low temperature resulted in Si precipitates of moderate size, which decreased the ductility [7]. However, for low-Mg alloy, either an increase in the number of Si precipitates or an increased coarsening of the silicon precipitates was expected, the effect of a reduced amount of hardening precipitates was “neutralized” by the increased amount of brittle silicon precipitates, therefore, the expected overall increased ductility wasn’t observed [7]. Also increasing silicon level above the amount required for stoichiometric formation of Mg_2Si was found to increase the strength of Al-Si-Mg alloys.

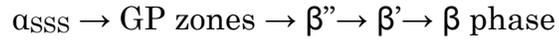
1.3 Effect of aging time and temperature

Age hardening has been recognized as one of the most important methods for strengthening aluminum alloys, which involves strengthening the alloys by coherent precipitates that are capable of being sheared by dislocations [46]. It

was indicated that aging must be accomplished below a metastable miscibility gap called Guinier-Preston (GP) zone solvus line [1]. Age hardening can take place either at room temperature (natural, T4 temper) or at elevated temperatures (artificial, T6 temper).

The phenomenon of precipitation was originally discovered by Alfred *Wilm* in 1906 [47]. He found the hardness of aluminum alloys that contained magnesium, copper, and other trace elements increased with time at room temperature, which was later explained by precipitation hardening. Over years lots of work was done to understand the aging kinetics of T4 and T6 heat treatments and to study the effect of under-aging, peak-aging, and over-aging on hardness [48-50], ultimate tensile strength [50], thermal fatigue properties [51], crack propagation behavior [52], and cyclic stress-strain response of cast aluminum alloy A356 [53]. *Li et al* [50] reported age-hardening behavior of cast aluminum alloy A356. At higher aging temperature peak hardness was obtained at shorter aging times since the diffusion was faster at higher temperature. Also various age hardening models, based on thermodynamics, kinetics, and dislocation mechanics, were developed for aluminum alloys in recent years. An age hardening model, based on the Shercliff and Ashby methodology, was developed by *Rometsch* and could be used to successfully predict the yield strength of cast aluminum alloy A356 aged for different times [54].

It is well accepted that the precipitation sequence responsible for age hardening of Al-Si-Mg alloys is based on the Mg_2Si precipitates and represented by the following stages [48, 50]:



where α_{SSS} stands for a supersaturated solid solution, GP zones are the Guinier-Preston zones, β'' and β' are the metastable phases, and β is the equilibrium phase.

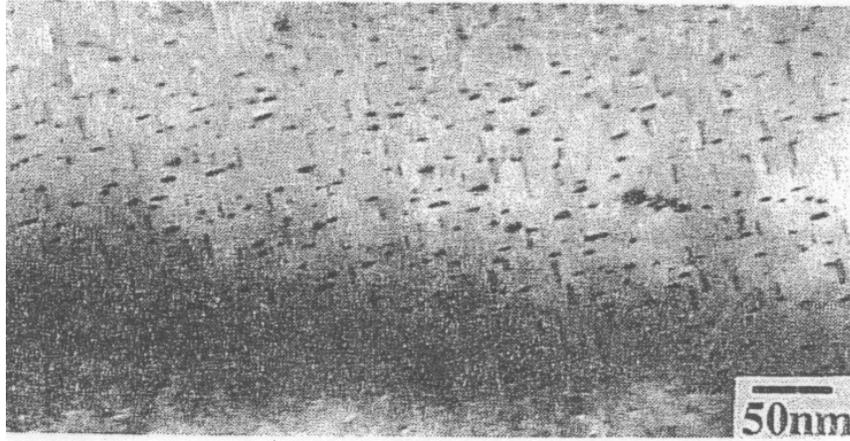


Figure 8. TEM micrograph showing β'' precipitates in the α -Aluminum matrix of A356 alloy solutionized at 540°C for 14 hours, 25°C water quench, and aged for 10 hours at 170°C. Beam direction: $\langle 110 \rangle_{Al}$ [40]

From TEM examination, *Zhang* observed that β'' precipitates exhibited a needle shape with the growth direction parallel to the $\langle 001 \rangle$ aluminum zone axis [40]. The size depended on the specific quenching and aging condition, e.g. 3-5nm in diameter and 10-20nm in length with water quench and aging at 170°C for 10 hours, as shown in Figure 8 [40]. However, other than the

precipitation of Mg_2Si , silicon precipitates in Figure 9, ranging 30-80nm in size, were observed both in the primary aluminum dendrites and in the eutectic region after the sample was aged for 24 hours. The number of silicon precipitates increased with the increase in aging time [40]. This phenomenon was due to the excess silicon in the matrix that wasn't needed for forming Mg_2Si precipitates [40, 55]. It was also found silicon precipitates required a longer incubation time, like 3-6 hours [56].

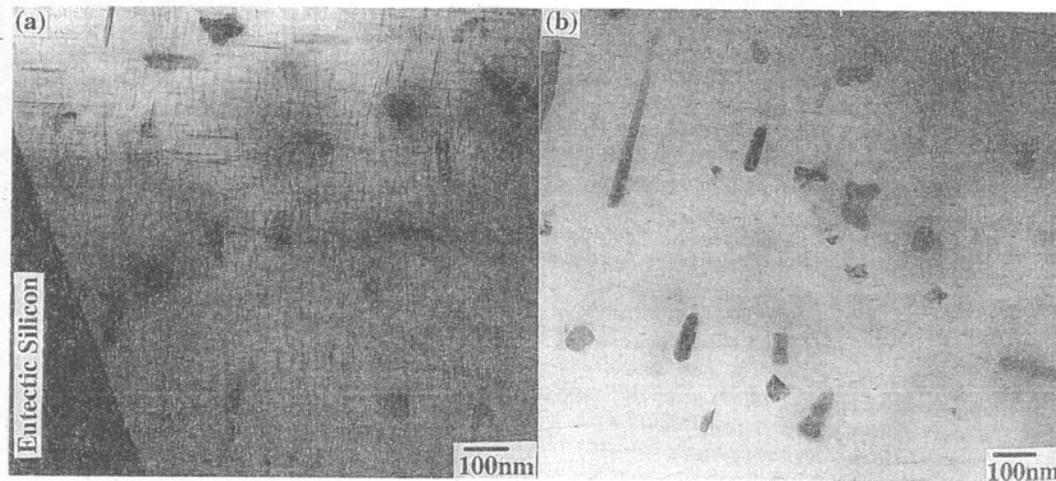


Figure 9. (a) and (b) TEM micrographs showing silicon precipitates in the central region of a α -aluminum dendrite and preferred distribution of silicon precipitates along dislocations. 25°C water quench and ageing for 24 hours at 170°C [40]

Other than the standard T4 and T6 aging treatments, some nonstandard aging processes were also investigated for cast aluminum alloy A356. *Bian et al* [57] reported the as-cast aging process, without solutionizing and quenching steps, could reduce the distortion and residual stress problems that might be caused by the quenching process. The results showed the

ultimate strength and elongation of cast aluminum alloy A356 upon as-cast aging treatment were close to that could be obtained from the standard solutionizing-quench-aging treatment. Moreover, the addition of trace elements, Sn, Ce, and Be, was found to enhance the as-aged mechanical properties of cast aluminum alloy A356.

Lee et al [58] studied the effect of pre-aging on precipitation hardening of Al-Si-Mg alloys. The experimental results indicated that the highest hardening rate was obtained from 95°C water quench combined with 60 minute natural aging prior to artificial aging. At a short artificial aging time, the tensile strength and hardness from 95°C water quench was found to be superior to that from 25°C and 60°C water quench. The addition of trace elements was believed to be capable of inhibiting the formation of precipitates and hence reducing the property loss from the delayed aging. Trace elements had higher binding energy with vacancies, which could effectively reduce the diffusion coefficient of solute atoms [59]. Therefore, clusters of precipitates couldn't form easily. *Murali et al* [59] concluded that trace additions, In, Cd, Sn, and Cu, inhibited the delayed aging in the order of being listed. Indium addition showed superior tensile strength, whereas Cd addition provided greater ductility.

2.0 Quench in water and polymer solution

Cold water used to be the dominant quenchant for heat treating aluminum alloys. However, in many cases, cold water quench produces unacceptable distortion due to high thermal gradients induced upon cooling [10, 11, 19, 20]. One of the earliest alternative method to cold water quench was “delayed quenching”. Fink and Willey reported the use of a delayed quenching process where the aluminum alloy was initially quenched in boiling water followed by a cold water quench at an appropriate time [60], but this method didn’t solve the quench uniformity problems inherent with water quench. Hot water was often another alternative quenchant [10]. However, distortion reduction was often insufficient with hot water quench or the design minimum physical properties might not be achieved, especially for the parts with a complex geometry. In such cases, aqueous polymer solutions were found to be able to effectively control the distortion/residual stress and achieve the quench uniformity [11, 13, 15-20].

Different types of polymer solutions have been used for this purpose, but they all track back to two different working principles [19]. By using polymer solution, the cooling rate from air cool to oil quench can be achieved. Polymer solutions are used to reduce the cooling rate of water by forming an insulating film on the workpiece’s surface. The comparison between water

quench and polymer quench is shown in Figure 10. The insulating film can be formed according to two different principles.

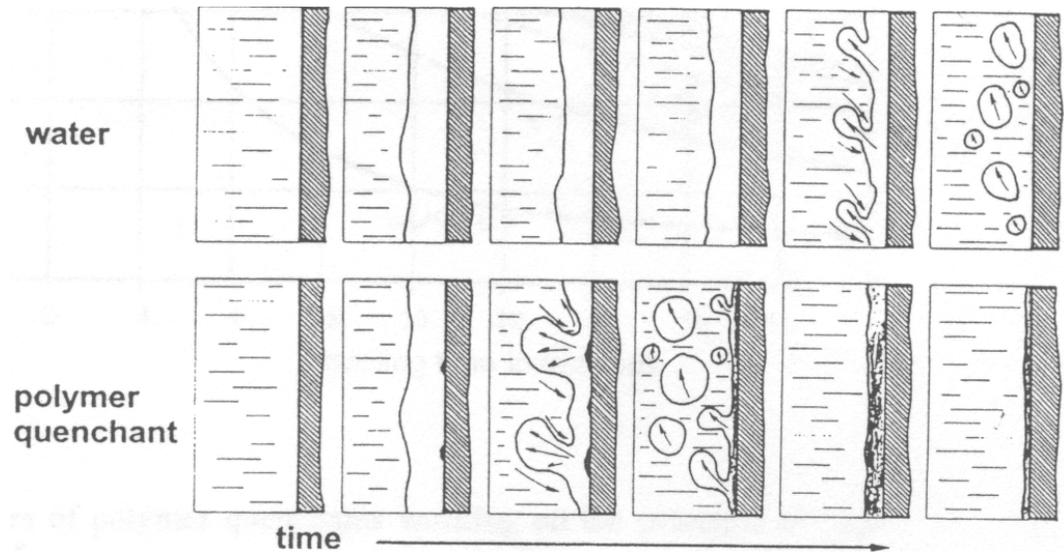


Figure 10. Schematic outline of the formation of polymer insulating films [19]

- (I) The first principle is often called “reverse insolubility/inverse solubility” [19], which means that the polymer is soluble in water at room temperature, but becomes insoluble when a certain temperature is reached. So when a part is quenched into polymer solution, due to the high temperature polymer condenses on the surface of the workpiece to form an insulating film, which can reduce the thermal gradient between the part and quenchant. When the temperature drops down to below the point of reverse

insolubility, the polymer dissolves back to the water. The thickness of film depends on the polymer type and concentration.

- (II) The second group of polymers forms the insulating film by the evaporation of water from part surface, thereby, leaving higher concentration of polymer on the surface. This is called “Film-forming by up-concentration” [19]. The film formed this way is very stable and effective in decreasing the cooling rate of water.

Extensive studies have been done on wrought aluminum alloys quenched in water and polymer solutions. Polymer quenchants formulated with a poly (alkylene glycol)-PAG copolymer, first reported by Blackwood and Cheeseman [61] and now designated as “Type-I“ polymer quenchant, have been used in the aluminum heat treatment industry for over 30 years as alternatives to hot-water quench for distortion control and crack prevention [62]. This kind of polymers is the copolymer of ethylene and propylene oxide [15]. Examples of aluminum alloys quenched in polymer solution can be found from the summary in references [62] [11, 20].

Other than aluminum alloys, polymer solutions also find a variety of applications in the heat treatment of steels since they are less expensive, cleaner, more flexible, and fire-resistant compared with the conventional quench oil. These applications include: production of saw blades, carburized

forged gears, pipe connectors, and truck crankshafts [15]; heat treating SAE 5160 automotive leaf springs [16]; quenching gears [17]; case hardening steels [13] and inverse hardening, intensive quenching, and immerse time quenching technology [18].

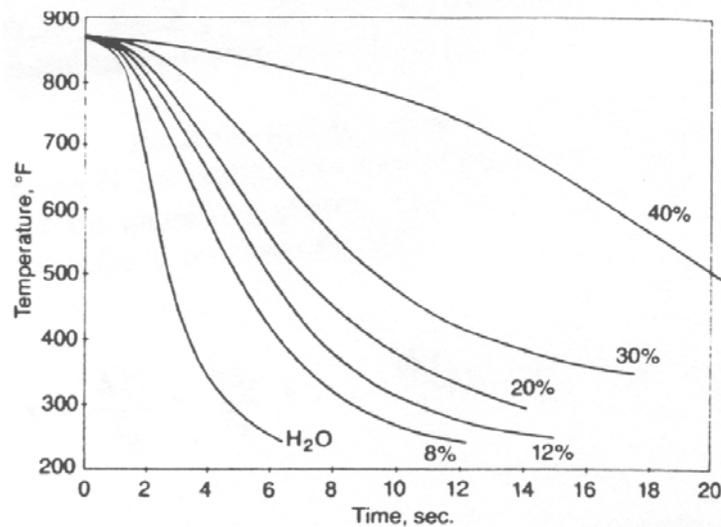


Figure 11. Cooling curves for a 1 inch 7075 aluminum alloy plate quenched into different concentrations of a Type I polymer quenchant [62]

For polymer quenchants, the quench uniformity is more crucial than the cooling rate itself. Polymer concentration and agitation play significant role on the quench uniformity and the attainable properties of aluminum alloys. The polymer film thickness depends on both agitation and concentration of a polymer solution. The heat transfer rate decreases as the thickness of polymer film increases. The time when the film breaks down is also

dependent on the film strength, concentration, bath temperature, and agitation.

In 1977, Torgerson and Kropp evaluated the effects of the concentration of UCON A on the physical property performance of 7050-T736 hand forgings [63]. The data showed the design minimums for 7050-T736 could be achieved easily with UCON A for plate sections up to 4.75 inch [63]. The effects of concentration of a PAG-based polymer solution on the cooling rate of wrought aluminum alloy 7075 were illustrated in Figure 11 [62]. The “rewetting” time was used to characterize the quench performance of this polymer solution at different concentration levels. It was observed that PAG-based polymer quenchants enhanced the quench uniformity of wetting by the formation of an insulating film, thus minimizing distortion. Increasing concentration level elongated the rewetting time, which was the time difference between the starting point of film boiling and the ending point of nucleate boiling.

Other than the concentration, the agitation is another important process parameter in polymer quench. *Hider* stated that, other than the volume flow induced from the agitation, the relative flow direction and the turbulence of the flow were also very important when the overall impact from agitation was assessed [64]. Four different laboratory agitation systems, Tensi system, H—baffle, J-tube, and ultrasonic system, were evaluated to identify their impact

on quenchant testing by cooling curve analysis [14]. Parameters like directionality, flow rate, and turbulence varied significantly from system to system although the propeller rotation was the same. Visually the agitation of fluid in Tensi system was more effective than the other systems, promoting more uniform fluid temperature during cooling [14]. Although it was generally assumed that water-based quenchants needed a high agitation rate, from years of practical operation *H. Beitz* believed that the flow-speed in a quench tank was sufficient when it allowed a good replacement of the heated quenchant near the part surface [19]. In terms of the flow direction, vertical reversing that could create more homogeneous motion of fluid should be preferred to horizontal movement.

Quantitative evaluation of the effects of each individual process parameter and their interaction, e.g. polymer concentration, agitation, on the cooling rate/ heat transfer coefficient of cast aluminum alloy A356 in PAG-based polymer solutions was not available in the literature. Also the impact of these process parameters on the as-cast and as-aged properties was not determined. In this study the Taguchi technique is employed to design the experiments for quantifying the effects from each individual process parameter on the overall quenching performance.

3.0 Quench Factor Analysis (QFA)

3.1 Background

Depending on the cooling rate during the quenching process, the precipitates heterogeneously nucleate at the grain boundaries or any available defects present in the α -Aluminum matrix. This kind of precipitation can result in the reduction in supersaturation of solid solution, which decreases the ability of an alloy to develop the maximum strength attainable with the subsequent aging treatment. In order to balance between properties and distortion/residual stress, quantitative measurement of the strength resulting from different cooling rates is needed for the quenching process design. Quench Factor analysis was developed to quantify the variation in strength due to cooling rates [9].

Fink and Willey [65] developed the first Time-Temperature-Property (TTP) C-curves for aluminum alloys. TTP curve for aluminum alloys is analogous to TTT diagram for steels. The amount of precipitation during quenching depends on how fast the alloy is cooled, which in turn determines the strength attainable with the subsequent aging treatment. Fink and Willey used the C-curves to predict the corrosion mode of 2024-T4 from different cooling rates [65]. It was found the specimens corroded by pitting attack at higher cooling rates and by intergranular mode at lower cooling rates. Using C-curves, Fink and Willey also determined that the critical temperature

range for the precipitation of 7075-T6 to occur was between 400°C and 290°C and they correlated the strength with the average cooling rate within this temperature range [9].

The work done by Fink and Willey was a milestone in the physical metallurgy of aluminum alloys, which illustrated the importance of quenching rates [9]. However, the average cooling rate method found its limitation in effectively predicting properties. Different quench paths with similar average cooling rate within the critical temperature range could end up with different properties. *Cahn* [66] noted that reactions involving nucleation and growth could be additive if the nucleation sites saturated early in the reaction and if the growth rate was only a function of the instantaneous temperature. For such additive reactions, he showed that a measure of the amount transformed during continuous cooling could be given by the following equation [9].

$$\tau = \int_{t_i}^{t_f} \frac{dt}{C_t(T)} \quad (1)$$

where τ is a measure of amount transformed; dt is the duration time at a temperature; t_i is the time at the start of a quench; t_f is the time at the end of a quench; $C_t(T)$ is the critical time for certain percentage of transformation from the TTP curve.

Based on Cahn's work, *Staley* [67] developed a model, called Quench Factor analysis, to predict the corrosion mode of 2024-T4. The assumptions used in this analysis include: the corrosion mode of 2024-T4 changes from pitting to intergranular when certain fraction of precipitation occurs, the precipitation reaction is isokinetic, and the fraction of precipitation can be summed up over a critical temperature range. Later *Evancho and Staley* [68] extended the concept of Quench Factor analysis to determine the effect of quench path on strength and hardness.

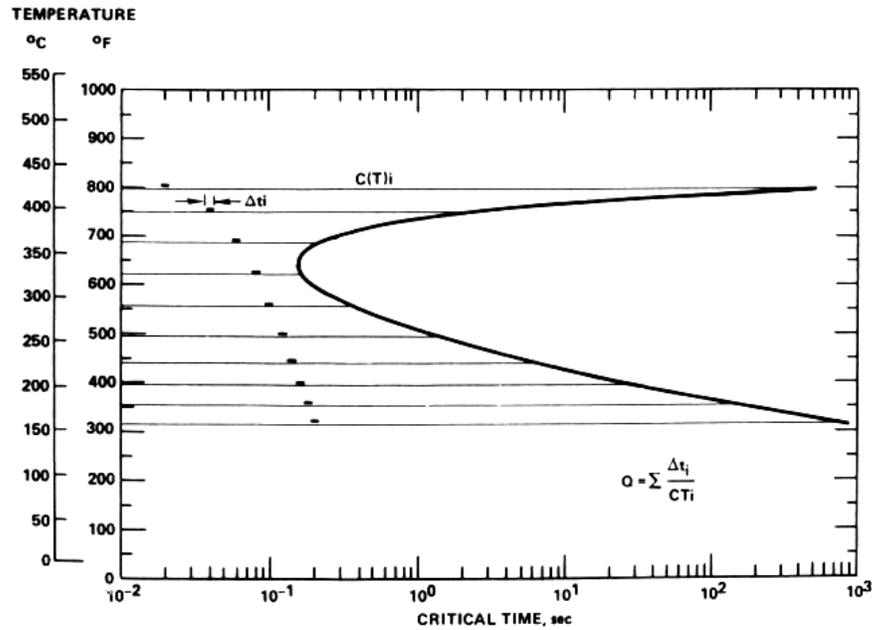


Figure 12. Schematic illustrations on plot of C_T function to calculate the Quench Factor [34]

Quench Factor analysis is a tool for predicting mechanical properties with a known quench path and the precipitation kinetics described by the Time-Temperature-Property (TTP) curve of an alloy. The advantage of this method

is that it provides a single number to correlate the cooling rate during quenching with the strength attainable from the subsequent aging. TTP curve in Figure 12 is a graphical representation of transformation kinetics that influences properties such as hardness or strength [34]. The assumptions behind the analysis are: The precipitation reaction during quenching is additive/isokinetic; the reduction in properties can be related to the loss of supersaturation of solid solution during quenching.

Quench Factor analysis can be illustrated as follows. The Quench Factor is typically calculated from a cooling curve and a C_T function, an equation that describes the transformation kinetics of an alloy. The C_T function was defined by Evancho and Staley and has the similar format as the reciprocal of the classical nucleation rate equation [9]. This function could be expressed using the following equation[9, 25, 27, 34, 69]:

$$C_T = -K_1 * K_2 * \text{Exp}\left[\frac{K_3 * K_4^2}{RT(K_4 - T)^2}\right] * \text{Exp}\left[\frac{K_5}{RT}\right] \quad (2)$$

C_T is the critical time required to form a constant amount of a new phase or reduce the strength by a specific amount; K_1 is a constant which equals the natural logarithm of the fraction untransformed during quenching (typically 99%: $\text{Ln}(0.99)=-0.01005$); K_2 is a constant related to the reciprocal of the number of nucleation sites; K_3 is a constant related to the energy required to form a nucleus; K_4 is a constant related to the solvus temperature; K_5 is a

constant related to the activation energy for diffusion; R is the universal gas constant, 8.3143 J/K*mole; T is the absolute temperature (K).

The incremental Quench Factor, q_f , is calculated for each time step on the cooling curve. q_f represents the ratio of the amount of time the alloy was at a particular temperature divided by the time required for a specific amount of transformation, typically 0.5% at a temperature [34].

$$q_f = \frac{\Delta t_i}{C_{T_i}} \quad (3)$$

where q_f is the incremental quench factor; Δt_i is the duration time at a temperature; C_{T_i} is the critical time required for certain fraction of precipitation to occur at a temperature.

The incremental Quench Factor are summed up over the entire transformation temperature range to produce a cumulative Quench Factor Q [9, 24, 29, 69]:

$$Q = \sum q_f = \sum_{T=M_s}^{T=A_r_3} \frac{\Delta t_i}{C_{T_i}} \quad (4)$$

Lower Quench Factor values are associated with rapid cooling and high attainable strength. The critical Quench Factor value is the maximum value that can result in the desired strength and this value can be defined in terms of the maximum amount of transformation during cooling [34]. With a known

Quench Factor, the as-aged strength can be predicted using the following equation [9, 24-26, 69]:

$$\frac{\sigma - \sigma_{\min}}{\sigma_{\max} - \sigma_{\min}} = \exp(-K_1 Q)^n \quad (5)$$

Quench Factor analysis has been applied to a wide range of wrought aluminum alloys to predict properties and/or optimize industrial quenching procedures [23, 26, 28, 29, 31-34, 70-73] . It has also been applied to steels and aluminum castings and is now recognized as an important technique for modeling property loss during continuous cooling [27].

The theoretical basis of Quench Factor analysis and how the Quench Factor analysis was used in solving industrial problems were reviewed by Staley [24]. Applications include the design of quench systems, the development of quench practices to optimize combinations of high strength and low residual stress/distortion, and predictions of magnitude of loss in strength as a result of unsuitable quenching conditions [24]. Quench Factor analysis was also extended to describe the rate of loss in toughness of an AA 6000 series aluminum alloys [24].

The Quench Factor model has been improved over years [24, 25, 74]. In the original version of model, σ_{\min} in equation (5) was assumed to be zero after long hold times at temperatures below the solvus. This assumption was

questioned since σ_{\min} would decrease with the decrease in temperature below the solvus [9]. A better approximation was made, which assumed that σ_{\min} was a constant independent of temperature [69, 74]. Based on this new assumption, the improved model was capable of accurately describing the loss of toughness and strength to a larger extent than the previous model for isothermal cooling. Although the model could successfully predict the loss of strength during continuous cooling, it provided a conservative overestimate of the loss of toughness [25].

Other than the controversy on σ_{\min} , *Rometsch et al* [69] also showed that the assumptions of the Avrami exponent in equation (5) being 1 and independent of the material weren't valid when compared with the experimental observation. It was suggested that transformation kinetics could be described more correctly by a modified Starink-Zahra equation with a physically realistic Avrami exponent of 1.5 or greater than by a Johnson-Mehl-Avrami-Kolmogorov type equation [69]. In order to further improve the Quench Factor model, the size, shape, and distribution of precipitates formed during continuous cooling and isothermal cooling need to be considered and correlated with the mechanical properties.

3.2 Quench Factor modeling

The development of Quench Factor analysis makes it possible to quantitatively determine the reduction in the attainable mechanical properties due to the heterogeneous precipitation during continuous cooling of aluminum alloys. However, in order to employ this analysis for the property prediction in the industrial practice, the transformation kinetics during quenching needs to be obtained for generating the Time-Temperature-Property (TTP) curves for the alloy of interest. These parameters are not available for most of cast aluminum alloys in the literature. Over years, a variety of methods have been used to estimate the kinetic parameters of wrought aluminum alloys during quenching.

Dolan et al. determined the kinetic parameters for 7175-T73 based on hardness, electrical conductivity, and tensile strength [32, 75]. The technique used in his study was the interrupted quench method developed by Fink and Willey [76]. The experiments were performed at 10 intermediate temperatures ranging from 190°C to 415°C with 25°C intervals for different periods of times. With the experimentally measured properties, the TTP curve was generated using least squares best fit method and the constants K_2 - K_5 were estimated by non-linear regression analysis [32]. He suggested that the accurate calculation of Quench Factor required the time step interval should be selected so that the temperature drop was smaller than

25°C [32], the same finding was reported by *Totten et al* [28]. In their investigation it was found that the selection of time step ranging from 0.1 to 0.4 seconds didn't cause any appreciable variation in the calculated Quench Factor while considerable scatter was seen when the time step used was between 0.5 and 0.8 seconds [28]. TTP curve for aluminum 7010 was obtained by *Flynn and Robinson* also with the interrupted technique in terms of tensile strength, hardness, and electrical conductivity. The kinetic parameters K_2 - K_5 were determined using multiple regression analysis [31]. The maximum property in their quench factor model was obtained after the sample was solutionized, quenched to room temperature and subsequently aged to the T76 condition. The minimum property was approximated as zero.

Staley gave an example of using Quench Factor analysis to design an extrusion quench system that could be used to quench the extruded shapes of AA 6061 as the materials left the die [24]. The cooling data was acquired using the delayed quench method. The kinetic parameters were estimated by an iterative procedure [24]. Appropriate values were first assigned to the constants K_2 - K_5 , with which the property was estimated and compared with the experimentally measured property. These constants were systematically adjusted until the sum of the squares of the difference between the estimated and measured property was minimized [24]. Zero value was used as the minimum property. The validation of Quench Factor technique was

performed by *Bernardin and Mudawar* [29]. The TTP curve for aluminum 2024 was established with the delayed quench technique in terms of Rockwell B hardness. The maximum and minimum hardness used in the model are 78.4HRB and 2.2HRB [29]. The model was verified by heat treating a complex L-shaped specimen. The predicted hardness agreed well with that experimentally measured.

Interrupted quench is a precise method to study the precipitation kinetics of aluminum alloys during quenching. However, this method requires a lot of experimental efforts and special apparatus to carry out the isothermal tests. One fundamental aspect of Quench Factor Analysis (QFA) is to be able to use the isothermal transformation kinetics to predict the amount of transformation during continuous cooling [69]. If the cooling curve can be represented with a series of isothermal steps, then the amount of transformation at individual isothermal steps can be summed up over a critical temperature range to obtain the amount of overall transformation during quenching. Based on the additivity rule, some attempts have been made to generate TTP curves with the continuous cooling data as well as to estimate the kinetic parameters.

Rometsch and Schaffer constructed TTP curves for sand cast Al-7Si-Mg alloys in terms of yield strength with the continuous cooling data [27]. The samples

were quenched in different temperatures of water and in air after being solutionized. The aging was performed at an elevated temperature. Time-temperature data was collected during quenching and the yield strength measurement was made for each heat treatment condition. With the cooling curves and the experimentally measured yield strength, multiple linear regression analysis was used to estimate the constants K_2 - K_5 [27]. The maximum T6 yield strength was obtained from the sample quenched in room temperature water and then aged at 170°C for 8 hours. The minimum property was obtained after the sample was slowly cooled in the fluidized bed for over 24 hours and aged.

The interrupted quench technique requires tedious experimental work, however, the application of the Jominy End Quench method for the Quench Factor analysis has been successfully developed and used by MacKenzie and Newkirk to determine the kinetic parameters of wrought aluminum alloys 7075 and 7050 [77, 78]. In part of this research, we followed the procedures of the Jominy End Quench technique developed by MacKenzie and Newkirk [35, 77, 78] in collecting the experimental data. The Jominy End Quench method was originally developed to determine the hardenability of steels [79, 80], but now it has been widely applied to obtain an enhanced insight into non-ferrous alloys [11, 77, 81, 82] since it can provide multiple sets of cooling curves only with one quench. Mackenzie and Newkirk established the model

for wrought aluminum alloys 7075 and 7050 based on Vicker's hardness [77, 78]. Hardness measurements were made at selected locations along a Jominy End Quench bar and continuous cooling data (T-t) was collected at the corresponding locations. The maximum hardness was taken as the average of the first few readings near the quench end and the minimum hardness was taken as zero. Using equations (4) and (5), non-linear equations were established with the T-t data and the experimentally measured hardness. By solving these equations simultaneously, TTP curve was generated. The kinetic parameters were estimated from fitting the TTP curve with the non-linear least squares routine.

Although many successful predictions were made in the literature using the properties other than strength, most often with hardness, caution has to be taken when any other properties except strength is used in the Quench Factor model. The classical Quench Factor model was established in terms of the variation of strength with the retained solute concentration. In some cases the linear relationship between strength and hardness may be obtained, but the difference in strain hardening can cause poor correlations [69]. This might be compensated with a well-established hardness-strength conversion with the difference in strain hardening considered [69].

Minimum strength is another important variable in Quench Factor modeling. Different values have been used in the literature, including zero, a constant, as well as a variable as a function of temperature. Assuming a zero value of σ_{\min} in the model can provide acceptable predictions when the property loss is less than 10% [9]. A constant σ_{\min} has improved the model to be capable of predicting the property loss up to 15% [25]. For aluminum alloys, the precipitation rate isn't only a function of temperature, but also a function of the amount of precipitates available at certain temperature. More accurate prediction can be made only when $\sigma_{\min}(T)$ is used as a function of temperature. However, for most of the alloys the assumption of σ_{\min} as a constant is adequate since only the prediction at high ratio of property loss is of the concern [69]. If the Quench Factor model is obtained with the continuous cooling data, then σ_{\min} can be defined as a constant from T6 heat treatment after a very slow quench [69].

Quench Factor analysis has been successfully used for property prediction for wrought aluminum alloys. However, this kind of data is scarce for cast aluminum alloys. In this study, the kinetic parameters for cast aluminum alloy A356 will be estimated using the Jominy End Quench method and multiple linear regression analysis. The results will be experimentally verified.

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Chapter III

Paper #1

***“The Effects of Polymer Concentration and
Agitation on the Quench Performance of
Polymer Quenchant Aqua-Quench 260”***

by Shuhui Ma, Md. Maniruzzaman, and Richard D. Sisson, Jr.

The Effects of Polymer Concentration and Agitation on the Quench Performance of Polymer Quenchant Aqua-Quench 260

Shuhui Ma, Md. Maniruzzaman, and Richard D. Sisson, Jr.

Center for Heat Treating Excellence

Materials Science and Engineering

Worcester Polytechnic Institute

Keywords: Heat treatment, Quenching, Cast Al-Si-Mg alloys, Polymer quench, Taguchi design, ANOVA analysis.

ABSTRACT

Statistically designed experiments have been performed to investigate the effects of polymer concentration and agitation on the quenching characteristics of cast aluminum alloy A356 in aqueous solutions of Aqua-Quench 260 using the CHTE quenching-agitation system. Three levels of concentration and agitation were selected for this investigation. The experiments were designed using Taguchi technique and the experimental results were analyzed with analysis of variance (ANOVA) based on the average cooling rate. It is found that average cooling rate dramatically decreases with the increase in polymer concentration. Agitation only enhances the average cooling rate at low and medium concentration levels.

From ANOVA analysis, the process parameter that affects the variation of average cooling rate most is the polymer concentration, its percentage contribution is 97%. The effects from the agitation and the interaction between polymer concentration and tank agitation appear to be insignificant. From the study of aging kinetics, it is seen that the micro-hardness of cast aluminum alloy A356 increases with the aging time to a peak value and then decreases for a prolonged aging time under all the heat treatment conditions. The increase in the polymer concentration lowers the attainable hardness for polymer quenched samples to some extent.

I. INTRODUCTION

For age-hardenable aluminum alloys, the goal of quenching is to suppress the precipitation of a secondary phase during quenching process without distortion and excessive residual stress. Quenching media commonly used for aluminum alloys include brine solution, water, and polymer solutions [1-3]. Cold water had been the dominant quenchant for heat treating aluminum alloys. However, in many cases, cold water quench produces unacceptable distortion or high residual stress due to high thermal gradients generated upon cooling [1, 2, 4, 5]. Polymer quenchants are advantageous because they can provide a more uniform quench by extending the vapor blanket phase to a lower temperature [6, 7]. They are more environmentally friendly and can still maintain similar quenching performance [6-9]. They are flexible in quenching because their concentration in water can be varied to obtain the desired cooling rates [8, 9]. Also, polymer quenchants reduce the risk of fire and make it easier to control the cracking and distortion that water can often cause [4-6]. Reasonable cost is also an advantage to using polymers solutions over using oils.

The physical properties of polymer quench bath directly affect the cooling rate of a quenched part. These properties include the type of quenchant, its temperature, concentration, agitation level, and bath temperature [1, 10, 11]. These parameters must be controlled to optimize the quenching process in

terms of alloy microstructure, properties, and performance. Some investigations have been carried out by the researchers to study the effect of water temperature and the concentration of polymer solution on the mechanical properties of wrought aluminum alloys [2, 12-14]. *Emadi et al* [15] reported that increasing the water temperature or using air quench reduced the cooling rate and increased the chance of precipitation of a secondary phase during quenching. Torgerson and Kropp evaluated the effect of the concentration of UCON A on the physical property performance of 7050-T736 hand forgings [16]. The time-temperature data was collected for the alloy quenched in the polymer solution with the concentration range of 0% to 40%. The “rewetting” time was used to characterize the quench performance of polymer solutions at different concentration levels. It was found that increasing the concentration level elongated the rewetting time [16]. More quench uniformity from polymer quench was also observed in his investigation.

Other than concentration, agitation is another important process parameter in polymer quench. *Hider* stated that, other than the volume flow induced from the agitation, the relative flow direction and the turbulence of the flow were very important when the overall impact from agitation was assessed [17]. Canale et al reported the parameters like directionality, flow rate, and turbulence varied significantly from system to system although the propeller

rotation was the same [11]. *H. Beitz* believed in terms of the flow direction vertical reversing, which could create more homogeneous motion of fluid, should be preferred to horizontal movement [5].

Polymer solutions are not only widely used in quenching the wrought aluminum alloys, but also finds a variety of applications in the heat treatment of steels [7] [8] [9] [10] [6]. However, such data is scarce for cast aluminum alloys in the literature and quantitative measurement of the effects from each individual process parameter is not available. Therefore, in this study the Taguchi technique is employed to design the test matrix for investigating the effects of agitation and polymer concentration on the cooling rate, mechanical properties, and aging kinetics of cast aluminum alloy A356 in a PAG-based polymer quenchant.

II. EXPERIMENTAL

A. *Materials*

The mechanical properties of cast aluminum alloy A356 are very attractive for many applications in military and aircraft industries since the silicon, as the major alloying element, can offer excellent castability, good corrosion resistance, and machinability. The presence of small amount of magnesium makes the alloy heat treatable. The mechanical properties of the alloy can be greatly improved by heat treatment (T4 or T6). Chemical modification dramatically alters the morphology of eutectic silicon particles and provides a wide range of properties. Cast aluminum alloy A356 with the chemical composition in Table I is selected for the present investigation. The alloy in this study is modified with 0.02% strontium. It is reported that the addition of 0.008% strontium is sufficient to change an acicular eutectic to a finely dispersed fibrous eutectic for non-modified A356 alloy [18].

Table I. Chemical composition of cast aluminum alloy A356 (wt%)

Si	Mg	Cu	Mn	Fe	Zn	Ti	Sr	Al
7.20	0.35	0.01	0.0026	0.125	0.01	0.13	0.02	Balance

B. *Sample preparation*

Among the major casting processes, permanent mold casting can provide better mechanical properties, smoother cast surface, less tendency for entrapped gas, and finer dendrite arm spacing and grain structure.

Aluminum A356 cylindrical bars, 1" (2.54cm) in diameter and 8" (20.32cm) in length, were cast in the WPI Metal Processing Institute Advanced Casting Laboratory. The bars were cast in a permanent cast iron mold. The casting mold was preheated to 427°C (800°F) in a GECO BHT30 furnace. About 40 lbs of A356 knuckles were melted in a MELLEN CC12 resistance furnace and cast into the pre-heated cast iron mold. Prior to casting, the melt was degassed using Argon gas for about 90 minutes. A rotary impeller was used to agitate the melt. The melt pouring temperature was kept constant at 800°C (1472 °F). Cylindrical specimens, 1" in diameter and 4" in length, were fabricated from the cast bars and used in this study. As-cast surface was used in the quenching.



Figure 1. A cylindrical specimen of cast aluminum alloy A356

C. Experimental apparatus

CHTE quench-agitation system in Figure 2 was used in this investigation, which consisted of a MELLEN tubular furnace MA#100038 for heating the specimens, agitation system, data acquisition system, and connecting rod-

coupling-probe assembly. A U-shaped tube in the quench tank was used to direct the flow. An impeller, for agitation purpose, was introduced to the tube from one end; specimens were quenched into the other end when they were ready. Different agitation levels were obtained by adjusting the rotating speed of the impeller.

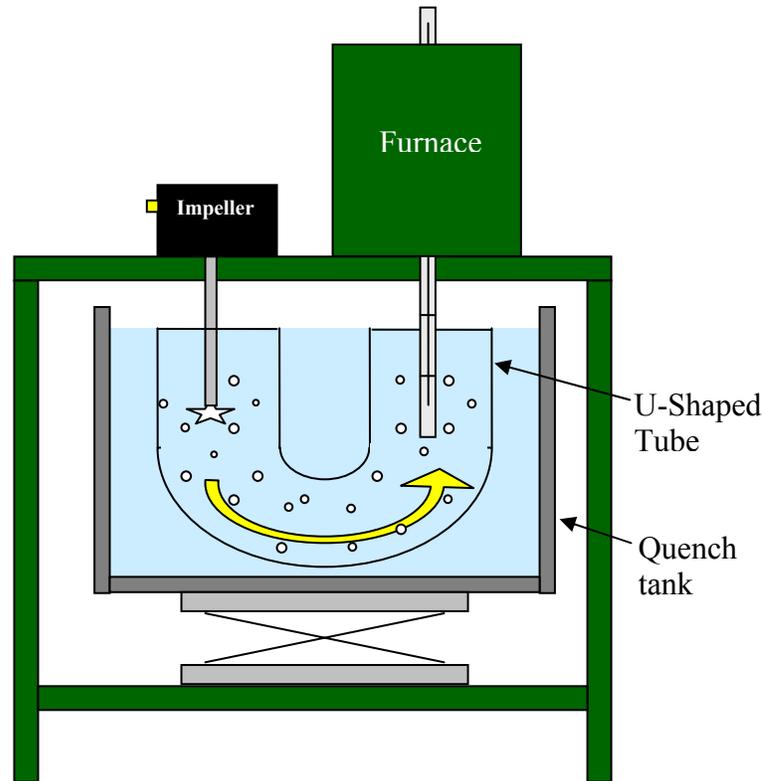


Figure 2. CHTE quench system

Specimens were solutionized at 540°C for 4 hours in a MELLEN tubular furnace MA#100038, quenched in water, polymer solution, and air at room temperature. The bars were then sliced into smaller disks and the individual pieces were aged at 165°C for 0, 2, 4, 6, 8, 10, 12 and 14 hours to study the aging kinetics. The time-temperature data was collected during the

quenching process using Labview VI 6.1. K-type thermocouples were placed in the geometric center of the specimens for this purpose. The collected data was smoothed by a running average method, an embedded algorithm in SigmaPlot (data analysis software). The first derivative of temperature in terms of time, called cooling rate, was taken to reveal the quenching stages and to compare the quench sensitivity of the alloy under different test conditions.

D. CFD simulation

The fluid field upon agitation was simulated using a numerical method called computational fluid dynamics (CFD) to visualize the magnitude and direction of the flow in the quench tank. CFD utilizes a computer model to solve the complex fluid flow that is often too difficult to solve with experimental or analytical techniques. There are many programs that use CFD to model fluid flow, in this study one specific program called Fluent¹ was used. The physical model and meshing of the quench tank were generated in Gambit and then imported into Fluent. The physical properties of fluid and materials and interfaces and boundary conditions were defined before the case was initiated. The program was run for a specified number of iterations. The iterative process was not completed until the convergence criterion was met. A residual plot was generated in Fluent to monitor the convergence of the

¹ ¹ Fluent, Inc. Web Site. 2002. [Online]. Available: <http://www.fluent.com/solutions/whatefd.htm>

case. In this study, two cases with different combinations of concentration and agitation were simulated. The test matrix is shown in Table II.

Table II. Parameters used in CFD simulation

	Concentration (%)	Agitation (rpm)
Case 1	10%	1300
	30%	1300
Case 2	20%	730
	20%	1950

III. RESULTS AND DISCUSSION

A. Taguchi design of experiments

Aqueous solution of a polymer has the advantage of providing more uniform quench over water by extending the vapor blanket stage to a lower temperature. In terms of polymer quench, there are two important parameters, which are the polymer concentration and the agitation applied to the quenchant. Quantitative measurement of the contribution from each process parameter to the heat extraction rate is a necessity for understanding the quenching process. Taguchi technique is employed for designing the test matrix to study these process parameters.

The velocity of a fluid attainable with the current agitation setup (impeller and U-shaped tube) was measured with a Turbo meter near one end of the U-shaped tube. In Figure 3 it shows that the velocity increases with the speed of impeller and remains constant after certain agitation level is reached. Beyond this level more turbulent flow is observed. Three agitation levels, labeled as low (0.5ft/sec), medium (1.5ft/sec), and high (2.5ft/sec) in Figure 3, were selected to be the input agitation levels in the Taguchi matrix. Another parameter of concern in Taguchi matrix is the polymer concentration. 10%, 20%, and 30% of polymer solution were chosen to be the levels of interest according to the recommendation from the manufacturer.

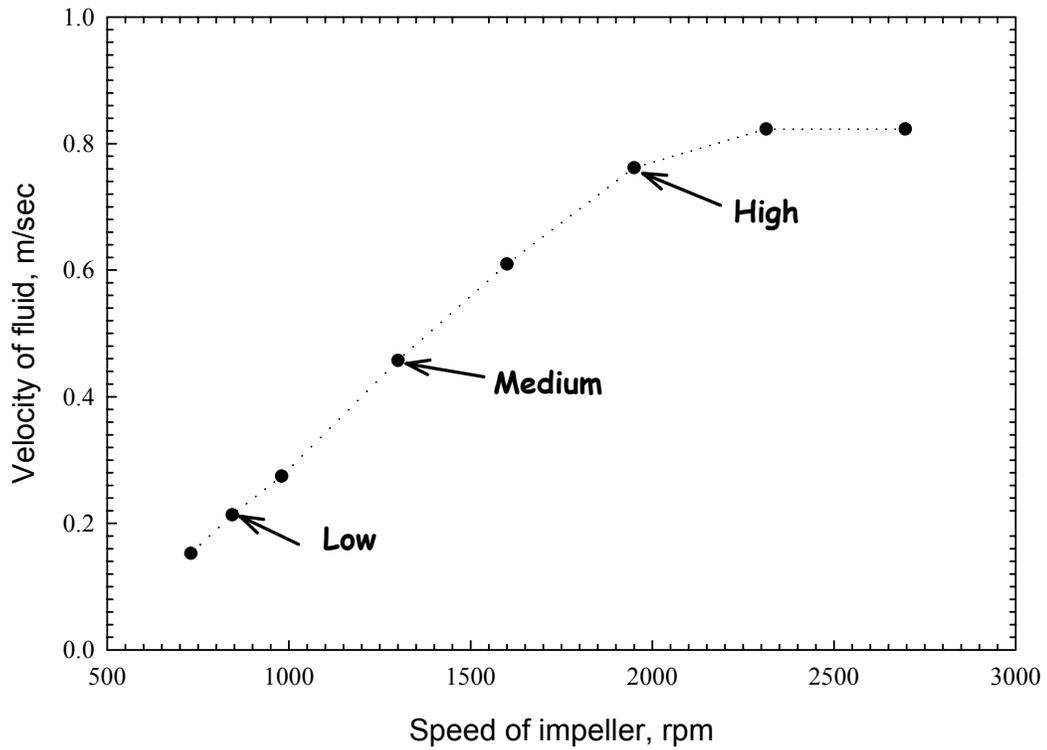


Figure 3. The variation of fluid velocity with the speed of impeller

Table III. Process parameters and the levels

<i>Factors</i>	<i>Level</i>		
	1	2	3
A Polymer concentration	10%	20%	30%
B Agitation, rpm	730 (Low)	1300 (Medium)	1950 (High)
Agitation, ft/sec	0.5	1.5	2.5

Two variables, each at three levels, were used in the matrix to study the heat transfer performance of cast aluminum alloy A356 in polymer solutions. Three-Level L9 orthogonal arrays were chosen to be the layout of DOE matrix. Table III summarizes the process parameters and the selected levels. Table IV shows the Taguchi L9 layout. “1, 2, 3” in Table IV stands for the

variable level in Table III. The percentage of effects from each variable and their interaction was analyzed by analysis of variance (ANOVA).

Table IV. Taguchi L9 Layout (Three-Level orthogonal arrays)

	Column No.			
Trial No.	1-A (Concentration)	2-B (Agitation)	3-A×B	4
1	1	1		
2	1	2		
3	1	3		
4	2	1		
5	2	2		
6	2	3		
7	3	1		
8	3	2		
9	3	3		

As shown in Table IV, nine tests of combination, three levels of concentration and three levels of agitation, were designed. Each condition was repeated for 3 times to produce the repeatability of the results. Figures 4 and 5 respectively shows the cooling rate curves of cast aluminum alloy A356 corresponding to different polymer concentrations and agitation levels. The small fluctuations on the plots were introduced from the vibration of the impeller. From Figure 4, the dramatic increase in the cooling rate with the increase in polymer concentration in the range of 10% to 30% is observed. The maximum cooling rate varies from 30°C/sec to 90°C/sec with the increase in polymer concentration by 20%. If the individual quenching stage is

examined, no much difference is seen in convection stage, but large variations are observed in the partial film boiling and nucleate boiling regimes.

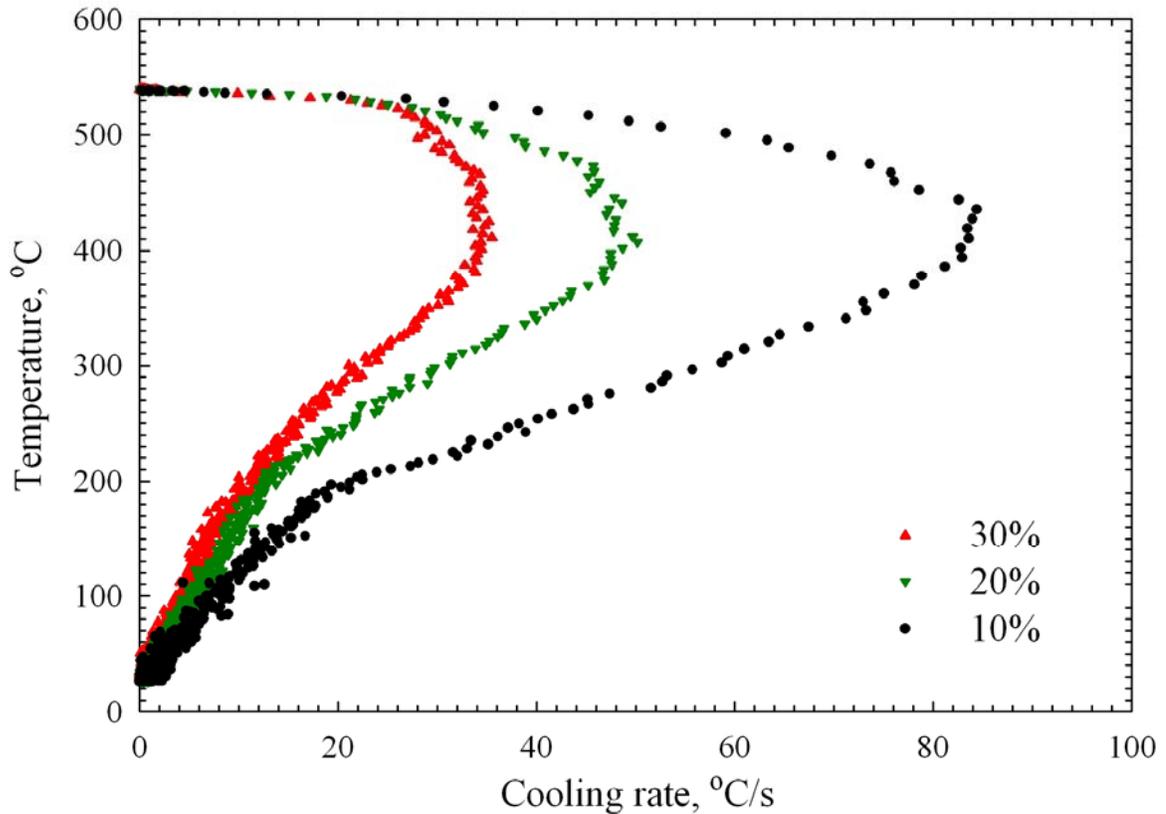


Figure 4. Cooling rate curves of Jominy End Quench bars of cast aluminum alloy A356 quenched in different concentrations of polymer solution with medium level of agitation (1300rpm).

In Figure 5, slight increase in cooling rate is seen when the agitation level increases from 730rpm to 1300rpm; however, further increase of agitation to 1900rpm does not introduce any increase to the cooling rate, instead the cooling rate drops. This phenomenon can be explained by the air bubble entrapment at high agitation level and the viscosity of the polymer solution. The entrapped air bubbles could visually be seen during the experiment.

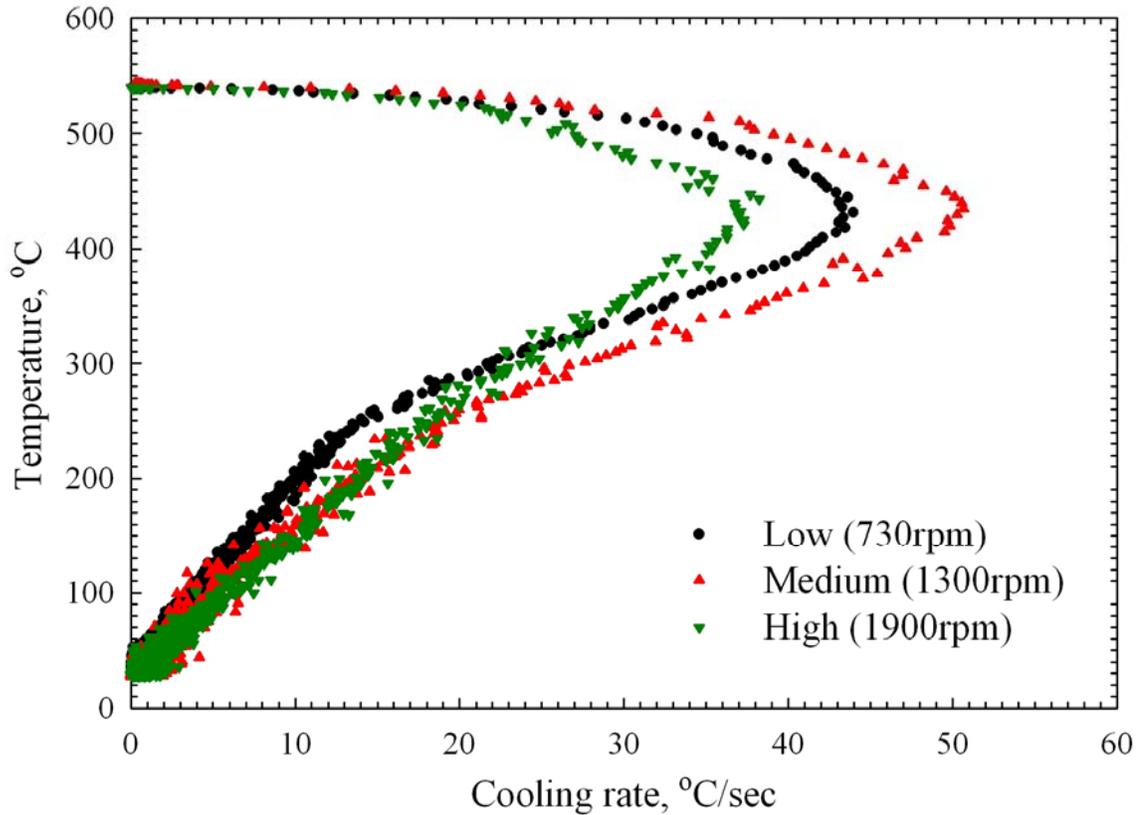


Figure 5. Cooling rate curves of Jominy End Quench bars of cast aluminum alloy A356 quenched in 20% Aqua 260 at different agitation levels.

The average cooling rate between 460°C and 280°C was chosen as the response variable for quantifying the effects of concentration and agitation. The selection of the temperature range is based on the CCT diagram of cast aluminum alloy A356 generated using JmatPro software and some reference data in the literature. The analysis of the experimental results is focused on maximizing the average cooling rate between 460°C and 280°C since this temperature range is critical for the precipitation of secondary phase, Mg_2Si , during the quenching of cast aluminum alloy A356. The variations of average cooling rate with the concentration and agitation are given in Figure 6. The

plot on the left side shows that average cooling rate slightly changes with agitation level for all three concentrations, while the plot on the right side reveals a dramatic drop in the average cooling rate with the increase in concentration.

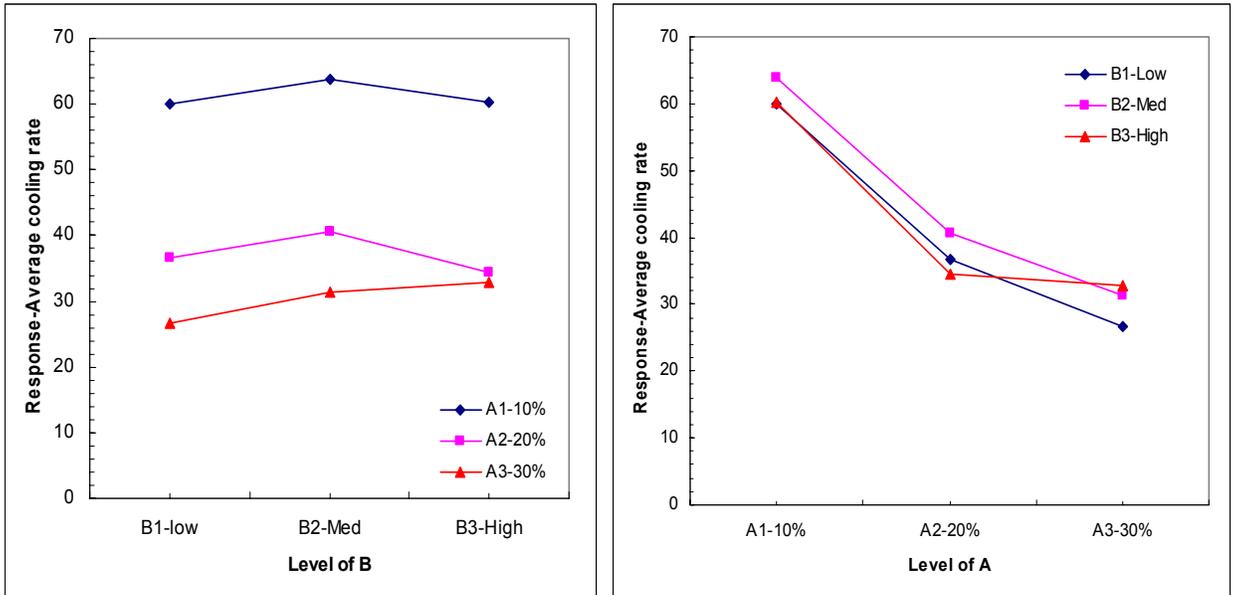


Figure 6. Variations of average cooling rate of cast aluminum alloy A356 with polymer concentration and tank agitation

B. ANOVA analysis

The goal of designing the experiments using the Taguchi technique is to optimize the experimental settings or process parameters for a multivariable process with least experimental efforts and to evaluate the experiment results with analysis of variance (ANOVA). In this study ANOVA was performed on the quenching data to quantitatively evaluate the effect from each process parameter and their interaction. Table V shows the results from this analysis. The percentage contribution reveals the relative effect from

each variable or their interaction. From Table V, the process parameter that affects the variation of average cooling rate most is the polymer concentration. The percentage of contribution from polymer concentration is 97%. The influences that the agitation and the interaction between concentration and agitation have on the average cooling rate are relatively insignificant. The same conclusion can also be drawn from Figure 6.

Table V. ANOVA analysis table for average cooling rate

Factors	Freedom	Sum of squares	Variance	Percentage of total effect
Factor A-concentration	<i>2</i>	<i>1590.15</i>	<i>795.08</i>	<i>97.66%</i>
Factor B-Agitation	<i>2</i>	<i>27.02</i>	<i>13.51</i>	<i>1.66%</i>
Factor A×B	<i>4</i>	<i>21.99</i>	<i>5.50</i>	<i>0.68%</i>
All other /error	<i>1</i>	<i>0.00</i>		
	<i>9</i>			
Total of sum of squares		<i>1639.17</i>	<i>814.09</i>	<i>100.00%</i>

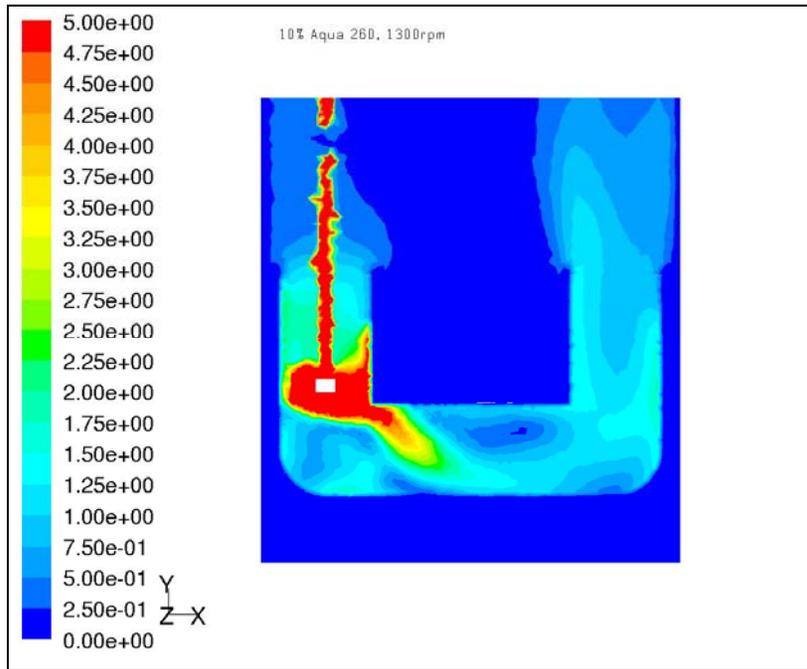
C. Fluent simulation

As shown in Table II, two cases were selected for CFD simulation to visualize the velocity distribution in the U-shaped tube and quench tank. The simulation was performed with the presence of impeller in the tube but without the quench probe. The viscosity and density of polymer solution was included in the boundary conditions of the model. Figure 7 gives the contour plot of velocity magnitude for two different levels of polymer concentration, 10% and 30%, upon the same agitation, 1300rpm. The color bar on the left

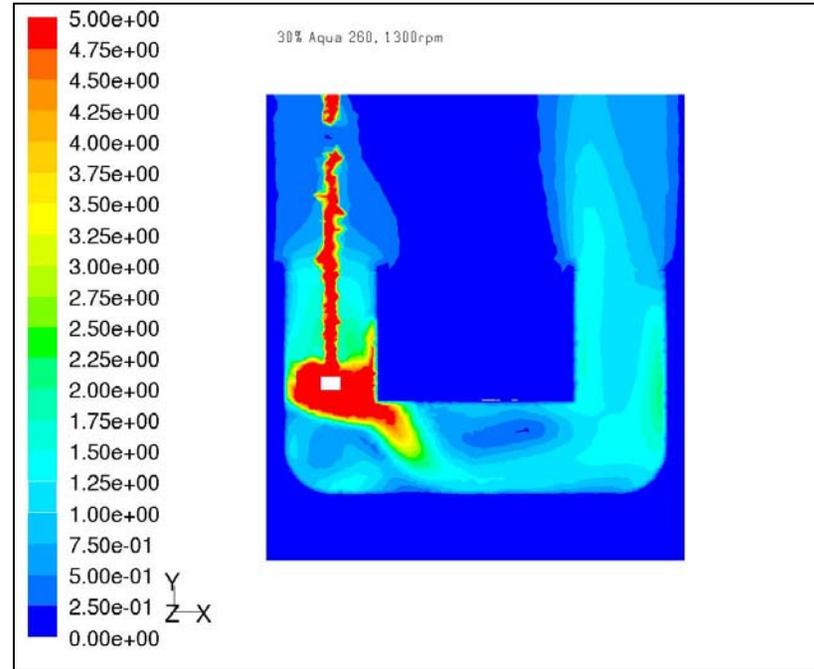
side of the plots indicates the velocity magnitude. Red color stands for the higher velocity and blue color represents lower velocity. The contour plot reveals the velocity distribution in the U-shaped tube. The same flow pattern is observed in Figure 7 for two different concentration levels upon the same agitation. Although a small dead zone is seen at the horizontal part of the tube, from the contour plot it can be seen the U-shaped tube does help direct the flow. Compared with the H-baffle used in the early stage of studying the agitation, U-shaped tube is more efficient. During the quenching tests, the quench probe was quenched into the same depth from the top surface of the U-shaped tube with the impeller on the other end. The magnitude of velocity at the location where the probe was quenched was extracted from the simulation data and listed in Table VI. Under the same agitation, velocity doesn't vary much with the variation in polymer concentration. However, from Figure 8 and the data in Table VI, if the polymer concentration is constant, increasing the agitation does increase the velocity to a great extent.

Table VI. Velocity magnitude from the Fluent simulation.

Polymer Concentration (%)	Agitation (rpm)	Velocity (m/s)
10%	1300rpm	1.0
30%	1300rpm	1.0
20%	730rpm	0.75
20%	1950rpm	2.5

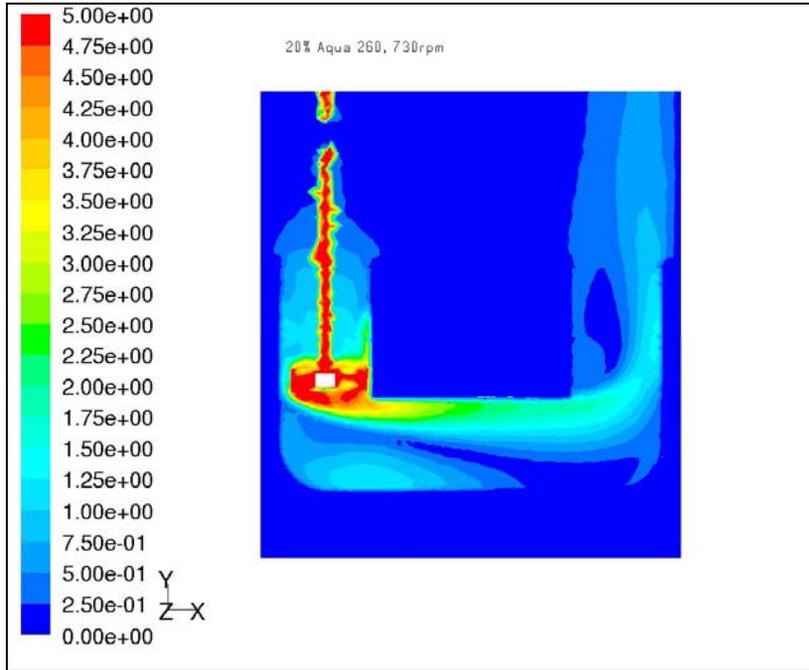


(a)

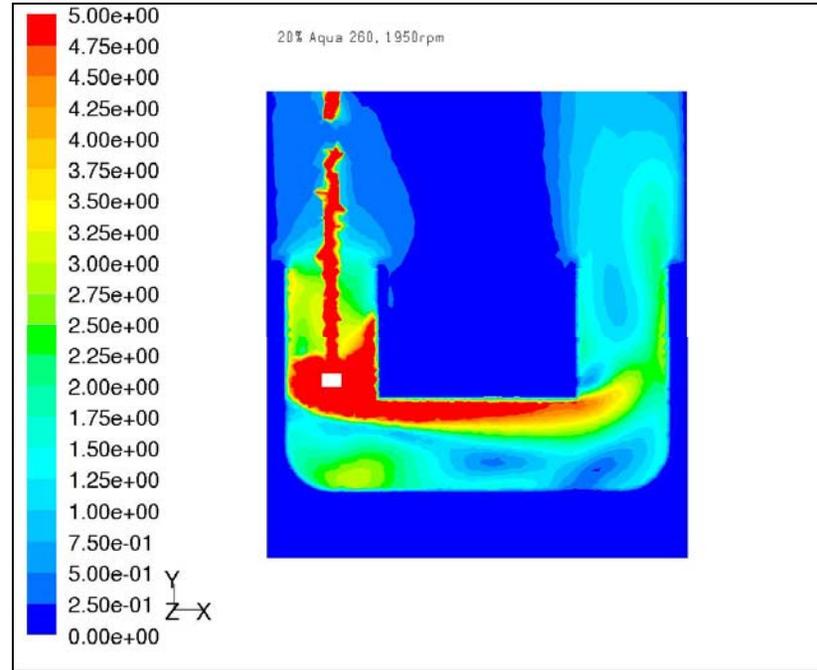


(b)

Figure 7. Contour plot of velocity field simulated in Fluent for case (a) 10% Aqua 260 (b) 30% Aqua 260 upon 1300rpm agitation.



(a)



(b)

Figure 8. Contour plot of velocity field in 20% Aqua 260 polymer solution simulated in Fluent for an agitation level of (a) 730rpm (b) 1950rpm

The velocity was measured near one end of the U-shaped tube with a Turbo meter. The fluid used in the measurement is water at room temperature. The simulated velocity with polymer solution at the same location was obtained from the velocity contour plot. The results are given in Table VII. Simulated and measured velocities are in the same range with the simulated one slightly higher.

Table VII. Simulated and measured velocity at one end of the U-shaped tube

<i>Polymer Concentration (%)</i>	<i>Agitation (rpm)</i>	<i>Measured velocity with water (m/s)</i>	<i>Simulated velocity (m/s)</i>
10%	1300	0.5	0.75
30%	1300		0.75 ~ 1.0
20%	730	0.2	0.25 ~ 0.5
20%	1950	0.8	1.0 ~ 1.25

D. Hardness measurements

The polymer concentration was determined to be the dominating process parameter from the above analysis of variance. The aging kinetics of cast aluminum alloy A356 was investigated after the specimens were solutionized and quenched in different concentrations of Aqua 260 polymer solution. The detailed test matrix is shown in Table VIII. The tank agitation used in this study is 1300rpm (medium level). After the solutionizing-quenching-aging process, the samples were grinded with SiC papers and polished with alumina down to 0.05 μ m. The micro-Vickers hardness measurements were

made on the cross section of the as-aged samples using Shimadu HMV-2000 with a load of 25gf and a dwell time of 10s. Ten readings were taken in the α -aluminum dendrites for each heat treatment condition; the average was used for comparison purpose. The samples were also quenched in water and air for comparison.

Table VIII. Test matrix for studying the aging kinetics of cast aluminum alloy A356

Solutionizing temperature	538°C							
Solutionizing time (hour)	4							
Quenching medium	Water, air, 10%, 20% and 30% Aqua-quench 260							
Aging temperature	165°C							
Aging time (hour)	0	2	4	6	8	10	12	14

Figure 9 showed the variation of micro-hardness of cast aluminum alloy A356 with aging times after the samples were quenched in different concentrations of aqueous solution of Aqua-quench 260, water, and air. Under all the heat treatment conditions, the micro-hardness increases with the aging time to a peak value and then decreases with a prolonged aging time. This can be explained by the evolution of Mg_2Si precipitates with the aging time and the interaction between the precipitates and dislocations.

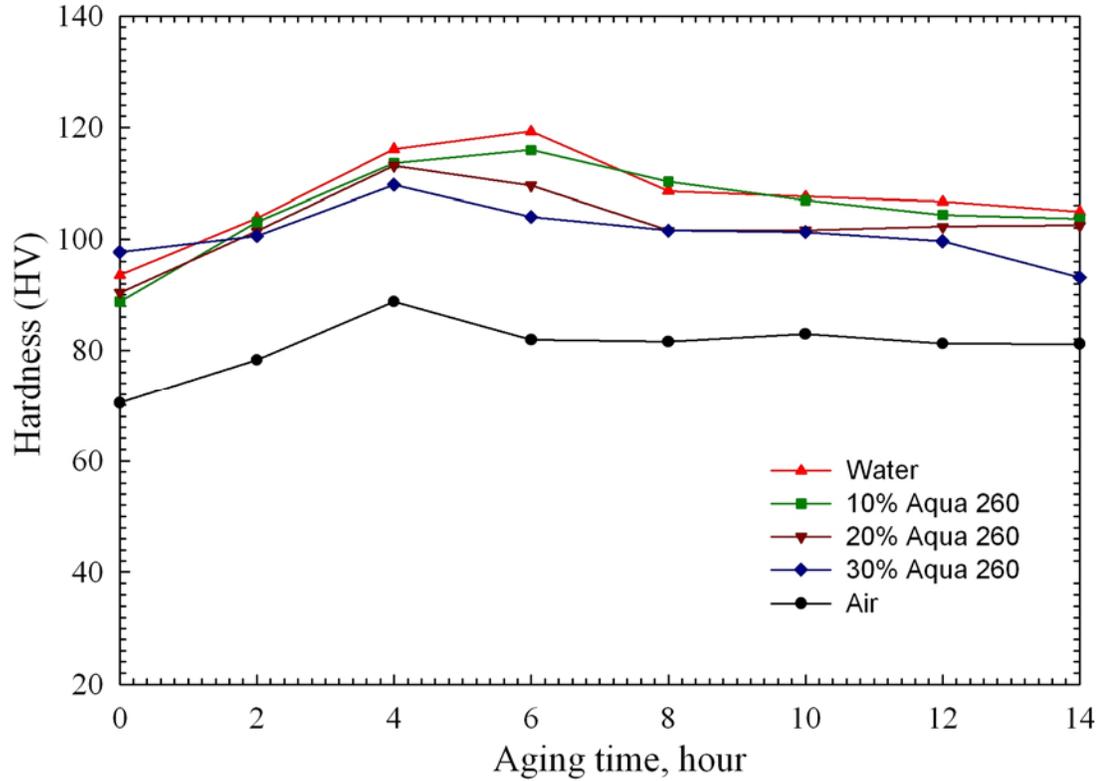


Figure 9. Vickers hardness of cast aluminum A356 solutionized, quenched in water, polymer solution and air, and aged at 165°C for different periods of times.

Water quenched samples show the highest hardness at all the aging times since they are subjected to the fastest cooling and relatively more supersaturated solid solution is retained to the room temperature. For all the polymer quenched samples, the increase in the polymer concentration lowers the attainable hardness if compared with the water quenched sample, as can be seen from Figure 9. However, the decrease in hardness due to the addition of polymer into water is not substantial. If the benefit, that more uniform quench could be achieved from using polymer solution, is considered, the data

in Figure 9 can provide the support to the advantages of using polymer to reduce the distortion/residual stress without sacrificing the property much. The similar result was reported by D.L.Zhang and L.Zheng in their study of quench sensitivity of cast Al-7%Si-0.4% Mg alloy [19]. They noticed the peak hardness did not change when the cooling rate decreased from 250°C/s to 110°C/s. It can also be seen from Figure 9 that the samples quenched in water and 10% polymer solution reaches the peak hardness at 6 hour aging, while the peak hardness is achieved at a shorter aging time (4 hours in this study) for the samples quenched in the 20%, 30% polymer solution, and air. As expected, slow air quench results in the lowest hardness, which is due to the reduction in retained solute concentration from the heterogeneous precipitation during quenching process.

IV. SUMMARY

The effects of process parameters, polymer concentration and agitation, on the quenching characteristics of cast aluminum alloy A356 in aqueous solution of Aqua-Quench 260 were investigated using the CHTE quenching-agitation system. The test matrix was designed with Taguchi technique and the experimental results were analyzed with analysis of variance (ANOVA) based on the average cooling rate.

1. The average cooling rate dramatically decreased with the increase in polymer concentration. Agitation only enhanced the average cooling rate at low and medium levels. When high agitation was employed, average cooling rate dropped.
2. From ANOVA analysis, the dominating process parameter that influenced the variation of average cooling rate was the polymer concentration; its percentage contribution was 97%. The effects from agitation and the interaction between polymer concentration and tank agitation appeared to be insignificant.
3. Under all the heat treatment conditions, the micro-hardness increased with the aging time to a peak value and then decreased with a prolonged aging time. Water quenched sample showed the highest hardness. The increase in the polymer concentration lowered the attainable hardness for polymer quenched samples. Air quench samples exhibited the lowest hardness as expected.

ACKNOWLEDGEMENTS

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Paper #2

***“A Methodology to Predict the Effects of
Quench Rates on Mechanical Properties of
Cast Aluminum Alloys”***

*by Shuhui Ma, Md. Maniruzzaman, D.S.MacKenzie,
and Richard D. Sisson, Jr.*

A Methodology to Predict the Effects of Quench Rates on Mechanical Properties of Cast Aluminum Alloys

Shuhui Ma¹, Md. Maniruzzaman¹, D.S.MacKenzie²,
and R.D. Sisson, Jr¹.

¹Center for Heat Treating Excellence
Materials Science and Engineering
Worcester Polytechnic Institute, Worcester. MA
²Houghton International, Valley Forge, PA

ABSTRACT

The mechanical properties of age-hardenable Al-Si-Mg alloys depend on the rate at which the alloy is cooled after the solutionizing heat treatment. Quench factor analysis, developed by Evancho and Staley, was able to quantify the effects of quenching rates on the as-aged properties of an aluminum alloy. This method has been previously used to successfully predict yield strength and hardness of wrought aluminum alloys. However, the Quench factor data for aluminum castings is still rare in the literature. In this study, the Jominy End Quench method was used to experimentally collect the time-temperature and hardness data as the inputs for Quench factor modeling. Multiple linear regression analysis was performed on the experimental data to estimate the kinetic parameters during quenching.

Time-Temperature-Property curves of cast aluminum alloy A356 were generated using the estimated kinetic parameters. Experimental verification was performed on a five-cylinder lost foam cast engine head. The predicted hardness agreed well with that experimentally measured. The methodology described in this paper requires little experimental effort and can also be used to experimentally estimate the kinetic parameters during quenching for other aluminum alloys.

I. INTRODUCTION

The heat treatment of aluminum alloys usually involves three steps: solutionizing, quenching, and aging. Depending on the cooling rate in the quenching process, precipitates can heterogeneously nucleate at the grain or phase boundaries or at any available defects present in the α -aluminum matrix. This kind of precipitation can result in reduction of supersaturation of the solid solution, which decreases the ability of the alloy to develop the maximum strength attainable with the subsequent aging treatment. A quantitative measurement of the strength resulting from different cooling rates is needed for the quenching process design [1]. Quench factor analysis, developed by Evancho and Staley, was able to quantify the variation in strength due to different cooling rates [1].

Quench factor analysis has been applied to a wide range of wrought aluminum alloys to predict properties and/or optimize industrial quenching procedures [2, 3], [4, 5]. It is now recognized as an important technique for modeling property variation during continuous cooling. In order to use quench factor analysis for property prediction, the kinetic parameters of an aluminum alloy during quenching need to be experimentally estimated and verified. Interrupted quench, developed by Fink and Willey [6], was traditionally employed by the researchers to collect the experimental data including the thermal history of the alloy being studied and the mechanical

properties from the corresponding quenching process. Using the interrupted quench technique, Dolan *et al.* determined the kinetic parameters for 7175-T73 based on hardness, electrical conductivity, and tensile strength [7, 8]. Staley gave an example of using quench factor analysis method to design an extrusion quench system that could be used to quench the extruded shapes of AA 6061 as the materials left the die [9]. Bernardin and Mudawar [10] generated the C-curve for wrought aluminum alloy 2024 with the delayed quench technique in terms of Rockwell B hardness.

The interrupted quench technique requires tedious experimental work, however, the application of the Jominy End Quench method for the quench factor analysis has been successfully developed and used by MacKenzie and Newkirk to estimate the kinetic parameters of wrought aluminum alloys 7075 and 7050 [5, 11]. The Jominy End Quench method was originally developed to determine the hardenability of steels [12, 13], but now it has been widely applied to obtain an enhanced insight into non-ferrous alloys [14-16] since it can provide multiple sets of cooling curves only with one quench.

There are a variety of ways to obtain C-curves and kinetic parameters with the experimentally measured properties and cooling data. C-curves of 7175-T73 were generated by Dolan *et al* using least squares best fit method and the constants K_2 - K_5 were determined with non-linear regression analysis [7].

Flynn and Robinson determined the kinetic parameters K_2 - K_5 for aluminum 7010 using multiple regression analysis [17]. Staley estimated the kinetic parameters of AA6061 with least squares routine [9]. MacKenzie and Newkirk [11] generated the C-curves of 7075 and 7050 by simultaneously solving a series of non-linear equations and the kinetic parameters were estimated by fitting the generated C-curve with the non-linear least squares routine.

However, the methodology for generating C-curves and kinetic parameters for cast aluminum alloys during quenching is not available in the literature. In this study, the experimental data was collected from the Jominy End Quench tests [4, 5]. Although generating C-curves by solving a series of non-linear equations and estimating K constants with non-linear least squares routine by MacKenzie and Newkirk have been successful [4, 5], this paper used multiple linear regression analysis [18] to estimate the kinetic parameters during cooling for cast aluminum alloy A356 with the experimentally collected data. The results were verified on a cast engine head [19]. The methodology described in this paper requires little experimental effort and can be used for estimating the kinetic parameters of other aluminum alloys, either cast or wrought.

II. THE MATHEMATICAL MODEL

Quench factor analysis is a tool for predicting mechanical properties of an alloy with a known quench path and the precipitation kinetics described by Time-Temperature-Property (TTP) curves. TTP curve in Figure 1 is a graphical representation of the transformation kinetics that influences such properties as hardness or strength [2]. The assumptions behind quench factor analysis are: the precipitation reaction during quenching is additive/isokinetic; and the reduction in strength can be related to the reduction of supersaturation of solid solution during quenching [9].

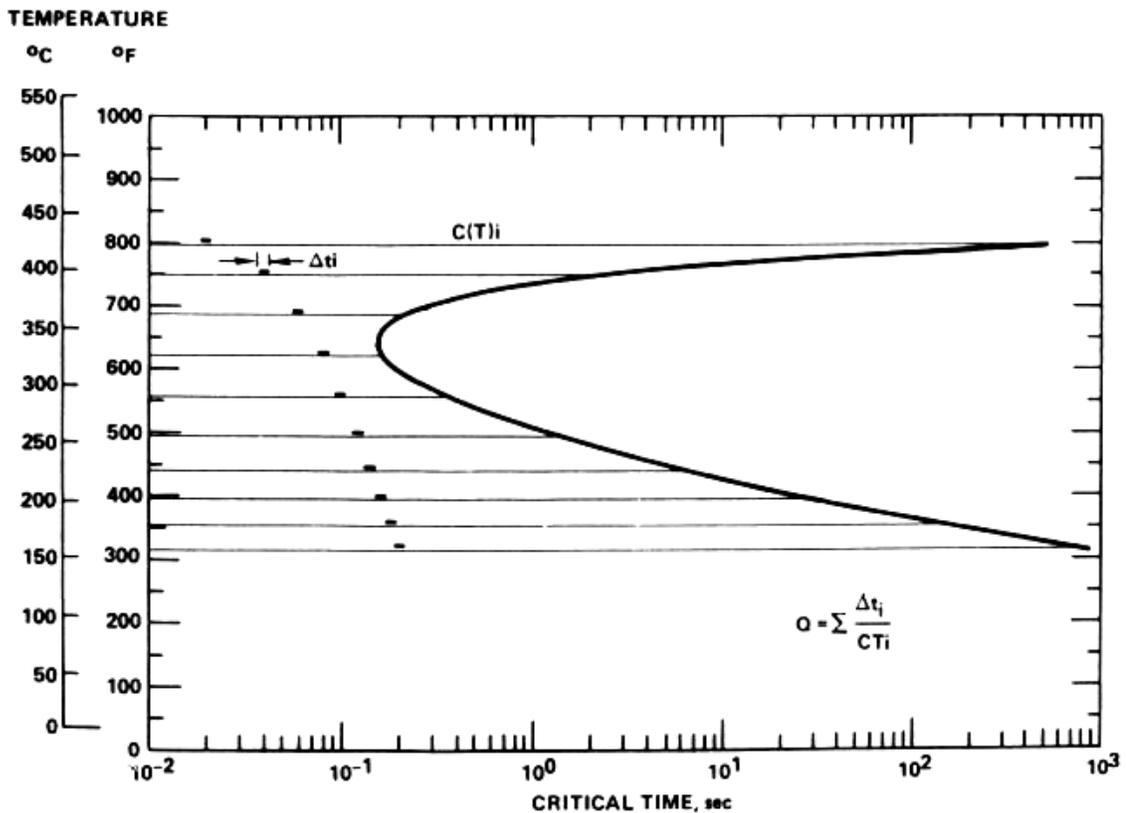


Figure 1 Schematic illustrations on plot of C_T function to calculate the quench factor [2].

The quench factor is typically calculated from a cooling curve and a C_T function, an equation that describes the transformation kinetics of an alloy. Evancho and Staley [9] defined the C_T function as having a form similar to the reciprocal of the nucleation rate equation. This form can be expressed using the following equation [1, 2, 5, 9]:

$$C_T = -K_1 * K_2 * \text{Exp} \left[\frac{K_3 * K_4^2}{RT(K_4 - T)^2} \right] * \text{Exp} \left[\frac{K_5}{RT} \right] \quad (1)$$

where, C_T is the critical time required to form a specific percentage of a new phase; K_1 is a constant which equals the natural logarithm of the fraction untransformed during quenching (typically 99.5%: $\text{Ln}(0.995) = -0.00501$); K_2 is a constant related to the reciprocal of the number of nucleation sites; K_3 is a constant related to the energy required to form a nucleus; K_4 is a constant related to the solvus temperature; K_5 is a constant related to the activation energy for diffusion; R is the universal gas constant, $8.3144 \text{ J}^\circ\text{K}^*\text{mol}$; T is the absolute temperature ($^\circ\text{K}$).

The incremental quench factor, q_f , represents the ratio of the amount of time the alloy is at a particular temperature divided by the time required for a specific amount of transformation [2]. The incremental quench factors can be calculated at each temperature and summed up over the entire transformation range to produce the cumulative quench factor Q [1, 9]:

$$Q = \sum q_f = \sum_{T_1}^{T_2} \frac{\Delta t_i}{C_{T_i}} \quad (2)$$

where q_f is the incremental quench factor and Δt_i is the time elapsed at a specific temperature.

With the calculated quench factor Q , the strength can be predicted using the following classical quench factor model [3, 9],

$$\frac{\sigma - \sigma_{\min}}{\sigma_{\max} - \sigma_{\min}} = \exp(K_1 Q)^n \quad (3)$$

where σ is the strength (In this study, σ represents the notation for Meyer hardness); σ_{\max} and σ_{\min} are the maximum and minimum strength achievable for a specific alloy; K_1 is decided above; n is the Avrami exponent.

Based on the classical quench factor model shown in Equation (3), improvements have been made to justify the assumptions for quench factor analysis, including the relationship between strength and solute concentration, minimum strength, and Avrami exponent [20]. The assumption of the linear relationship between strength and retained solute concentration was found to contradict the strengthening theory. According to the strengthening theory, Equation (3) is re-written as the following improved formula [20],

$$\frac{\sigma - \sigma_{\min}}{\sigma_{\max} - \sigma_{\min}} = \left[\exp(K_1 Q)^n \right]^{1/2} \quad (4)$$

This statement was justified by the morphology of the secondary phase that could precipitate out from the solid solution during quenching process.

A variety of mechanical properties have been used for quench factor modeling, including Vickers hardness [4, 5, 7, 20], Rockwell hardness [2, 10], electrical conductivity [7, 17], yield strength [3, 20, 21], and tensile strength [7]. Although many successful predictions were made in the literature, the classical quench factor models were established based on the variation of strength with the retained solute concentration and caution has to be taken when any properties other than strength are used in the quench factor modeling unless a linear relationship between the strength and the property exists for the alloy being studied [20]. In this investigation, the Meyer hardness, \bar{P} , is the property used in the quench factor modeling, which has an approximately linear relationship with strength. The Meyer hardness is defined as [22],

$$\bar{P} = \frac{4L}{\pi d^2} \quad (5)$$

Where \bar{P} is the Meyer hardness, MPa; L is the load, Kg; d is the diameter of indentation, mm.

The relationship between Rockwell hardness and Meyer hardness can be experimentally determined for any specific alloy. For cast aluminum alloy A356, the conversion was established by Tiryakioglu and Campbell using regression analysis of the experimental data [22]. The indentation size, d, is correlated with Rockwell B hardness in the following description [22],

$$d = 1.263 - 5.270 \times 10^{-3} RHB \quad (6)$$

Using Equations (5) and (6), the Meyer hardness can be calculated from the experimentally measured Rockwell hardness in B scale. The reason of using the Meyer hardness in the quench factor modeling is because it has a linear relationship with strength so the assumptions for quench factor models are still valid in this case.

III. RESEARCH METHODOLOGY

The research methodology used in this paper for estimating the kinetic parameters of aluminum alloys during quenching, is illustrated in Figure 2. This methodology starts from preparing an aluminum alloy of interest and casting the Jominy End Quench bars. Based on the ASTM standard A255, the Jominy End Quench tests are performed to experimentally collect time-temperature and Rockwell hardness data at selected locations on a bar. The advantage of using Jominy End Quench method for quench factor modeling is that a large range of cooling rates can be obtained with only one quench, which dramatically reduces the experimental efforts that are usually required with any other method. Rockwell hardness is converted to the Meyer hardness using the relationship established by Tiryakioglu and Campbell, as shown in Equations (5) and (6) [22]. Multiple linear regression analysis is performed on the experimental data to numerically estimate the kinetic parameters. These kinetic parameters are experimentally verified on a cast engine cylinder head. This methodology requires little experimental effort,

has been illustrated for cast aluminum alloy A356, and can be used to experimentally estimate the kinetic parameters during quenching for other heat-treatable aluminum alloys. More detailed procedures of this methodology are in the “results and discussion” section.

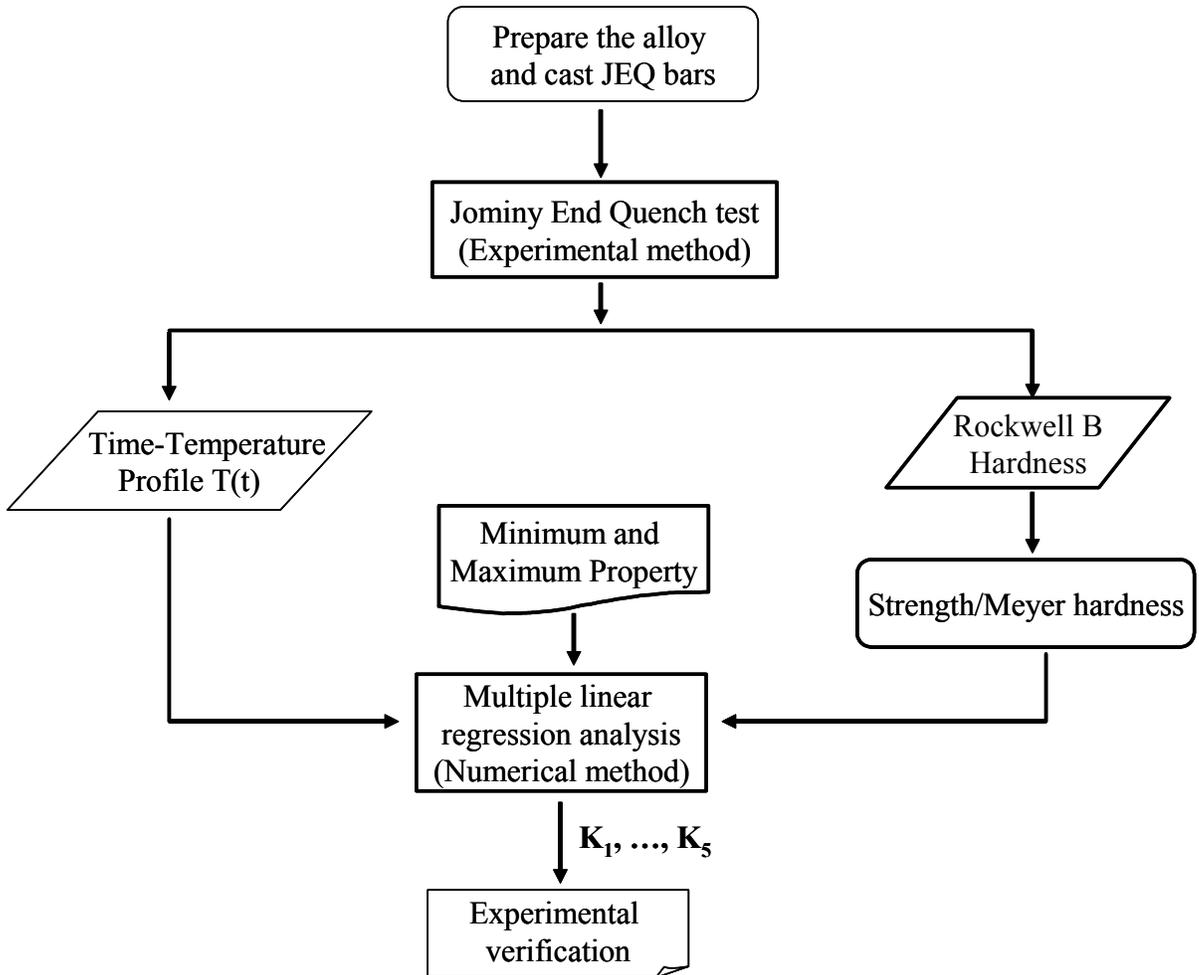


Figure 2 Overview of the research methodology for quench factor analysis.

IV. EXPERIMENTAL

A. Materials and Sample preparation

Aluminum A356 cylindrical bars with 2.54cm in diameter and 20.32cm in length were cast in the WPI Metal Processing Institutes Advanced Casting Laboratory. The bars were cast in a permanent cast iron mold. The casting mold was preheated to 427°C (800°F) in a GEICO BHT30 furnace. About 40lbs of A356 aluminum alloy was melted in a MELLEN CC12 resistance furnace and cast into the pre-heated cast iron mold. Prior to casting, the melt was degassed using Argon gas for about 90 minutes. A rotary impeller was used to agitate the melt during degassing. The melt pouring temperature was kept constant at 800°C (1472 °F) in the furnace. Jominy End Quench specimens (2.54cm in diameter, 10.16cm in length), as shown in Figure 3, were fabricated from the cast bars according to SAE J406 and ASTM A255 standards. The chemical composition of cast aluminum alloy A356 used in this study is given in Table I. This alloy is modified with 0.02% strontium.



Figure 3 Dimension of a Jominy End Quench bar of cast aluminum A356

Table I. Chemical composition of cast aluminum alloy A356 (wt%)

Si	Mg	Cu	Mn	Fe	Zn	Ti	Sr	Al
7.20	0.35	0.01	0.0026	0.125	0.01	0.13	0.02	Balance

B. Experimental apparatus

The Jominy End Quench apparatus was built according to the standard described in the SAE J406 and ASTM A255 specifications. The schematic of the apparatus is shown in Figure 4. An orifice with 12.7mm in diameter is connected to the waterline through a plastic pipe for quenching. The top plate supports the part in position. According to the standards, the distance between the test specimen and the orifice is 12.7mm. Since the quenching occurs at one end of a bar, the cooling along an entire Jominy End Quench bar is one-dimensional.

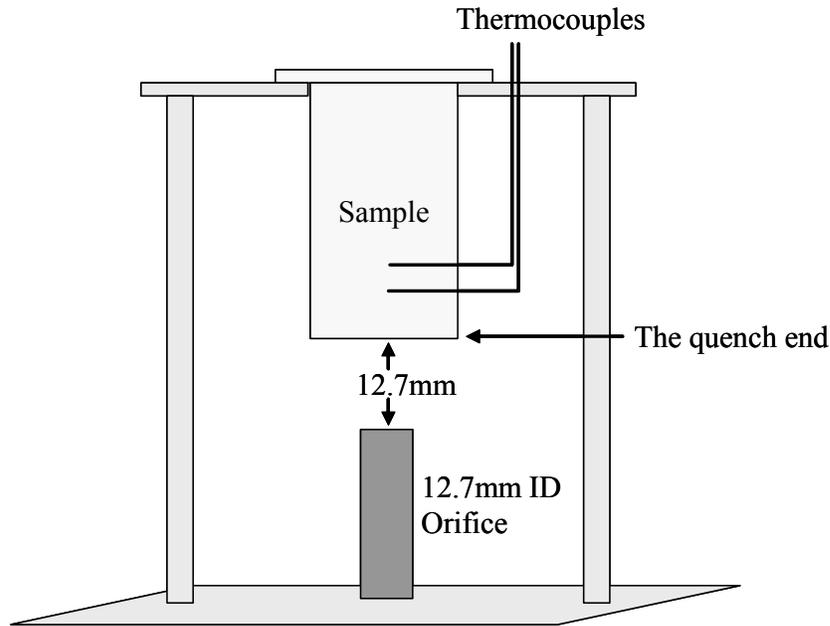
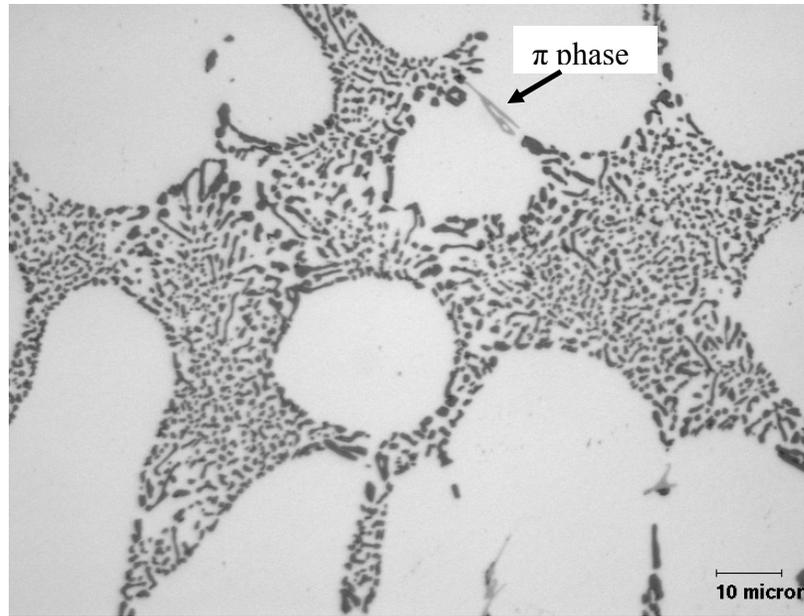


Figure 4 Schematic of the Jominy End Quench apparatus.

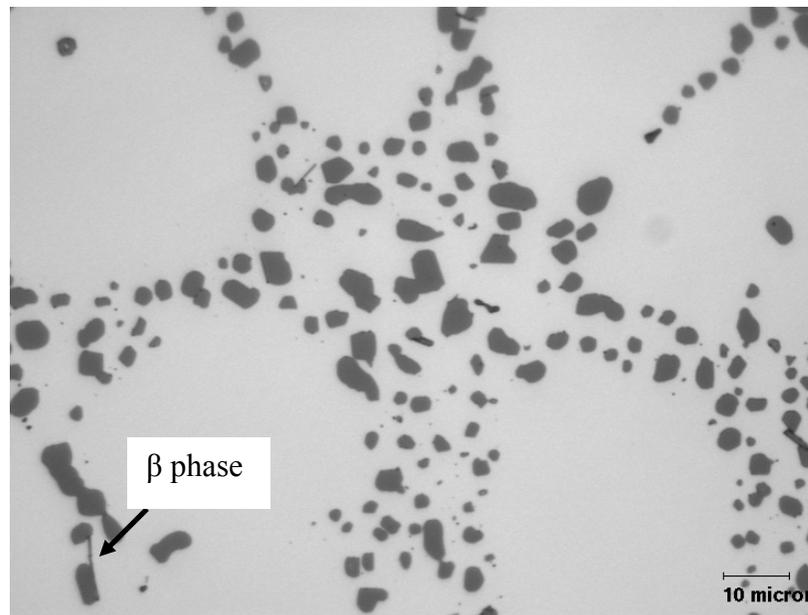
V. RESULTS AND DISCUSSION

A. Microstructure of cast aluminum A356

The microstructure of as-cast and as-solutionized cast aluminum alloy A356 was examined with both scanning electronic microscope and optical microscope. The shape and size of silicon particles reveal the extent of solutionizing. Solutionizing for long periods modify the morphology of the eutectic silicon. The rounding of silicon particles can effectively improve the ductility and the fatigue properties of the alloy. From Figure 5, both the spheroidisation of acicular silicon and coarsening of small silicon particles can be observed by comparing the silicon morphology before and after the solutionizing treatment. More spherical particles are seen in the as-solutionized sample that was solutionized at 540°C for 4 hours. Average equivalent diameter of Si particles in an as-solutionized Jominy End Quench bar is 3.6 μm . Fe-containing π/β phases can also be seen on the cell/grain boundaries. These iron-rich phases are detrimental to the materials and require a much longer solutionizing time to be dissolved. In most cases, complete dissolution of iron-containing phases is not observed [23].



(a) as-cast



(b) as-solutionized

Figure 5 Microstructure of (a) as-cast and (b) as-solutionized cast aluminum alloy A356

To determine the secondary dendrite arm spacing (SDAS) of the aluminum casting in this study and to verify there is no solidification gradient along cast bars, quantitative image analysis was performed at different locations of an as-solutionized Jominy End Quench bar by line intersection method. The magnitude of SDAS is an indication of solidification rate during casting process. SDAS is also an important parameter for estimating the solutionizing time needed for a cast aluminum alloy since it gives the range of the diffusion field for the diffusion of silicon, magnesium, manganese, and other addition elements during the solutionizing treatment in the case of cast aluminum alloy A356. Average size of SDAS for a cast aluminum alloy A356 bar in this study is 27 μ m and no variation is seen along the entire bar. The results are based on 10 measurements and shown in Figure 6.

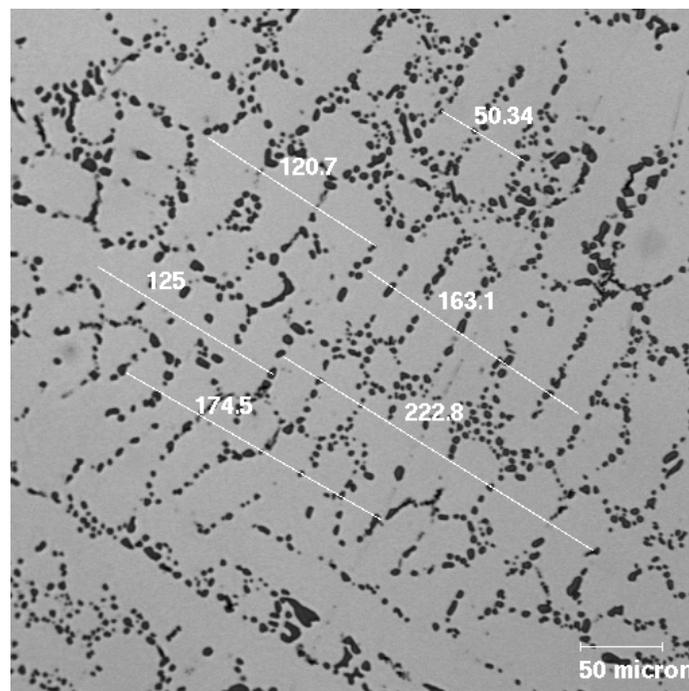


Figure 6 Measurement of SDAS of as-solutionized cast aluminum A356

Table II. Test matrix to study the effect of solutionizing/aging time on the hardness of A356

solutionizing temperature	538°C				
Solutionizing time (hour)	2	4	6	8	10
Aging temperature	165°C				
Aging time (hour)	6				
solutionizing temperature	538°C				
Solutionizing time (hour)	6				
Aging temperature	165°C				
Aging time (hour)	2	4	6	8	10

B. Effects of solutionizing and aging time

From an energy savings point of view, research has been focusing on examining the possibility of shortening the heat treatment cycle, especially reducing the solutionizing and aging time without sacrificing mechanical properties to a great extent. In this study, the first set of experiments was designed to characterize the effect of solutionizing time on the hardness of as-aged cast aluminum alloy A356 with other heat treatment parameters kept constant. The test matrix is given in Table II. The Rockwell B hardness measurements were made on the two flats along the bar, milled down 0.381mm from the surface, according to the ASTM standard. The results in Figure 7 show that the hardness drops gradually as the distance from the quench end increases. This phenomenon is due to the reduction in cooling rate along the Jominy End Quench bar, which decreases the retained

supersaturation of solute available for the subsequent aging treatment. The hardness from 2 hour solutionizing is the lowest and much lower than that for the samples solutionized at the same temperature for 4, 6, 8, and 10 hours. Only a small variation in hardness is observed when solutionizing time is greater than 4 hours. This finding agrees with what was reported in the literature, so it may be concluded that 4 hours is sufficient time for solutionizing cast aluminum alloy A356 with a SDAS of approximately $27\mu\text{m}$.

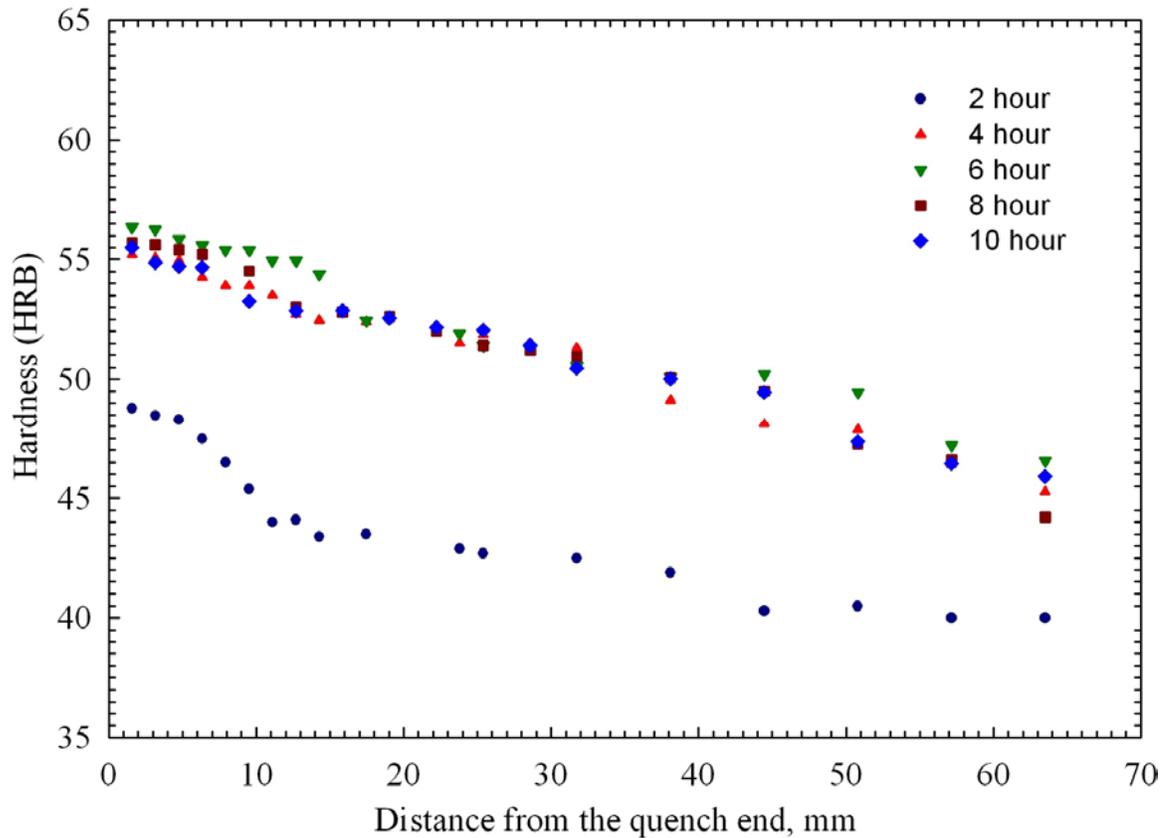


Figure 7 Hardness profile of a Jominy End Quench bar of cast aluminum alloy A356 with different solutionizing times.

It is well accepted that the precipitation sequence responsible for age hardening of Al-Si-Mg alloys is based on the Mg_2Si precipitates and represented by the following stages: α_{SSS} (α supersaturated solid solution) \rightarrow GP zones $\rightarrow \beta'' \rightarrow \beta' \rightarrow \beta$ phase [24, 25]. The strength of the alloy is determined by the size and distribution of precipitated particles as well as the coherency of the particles with the aluminum matrix.

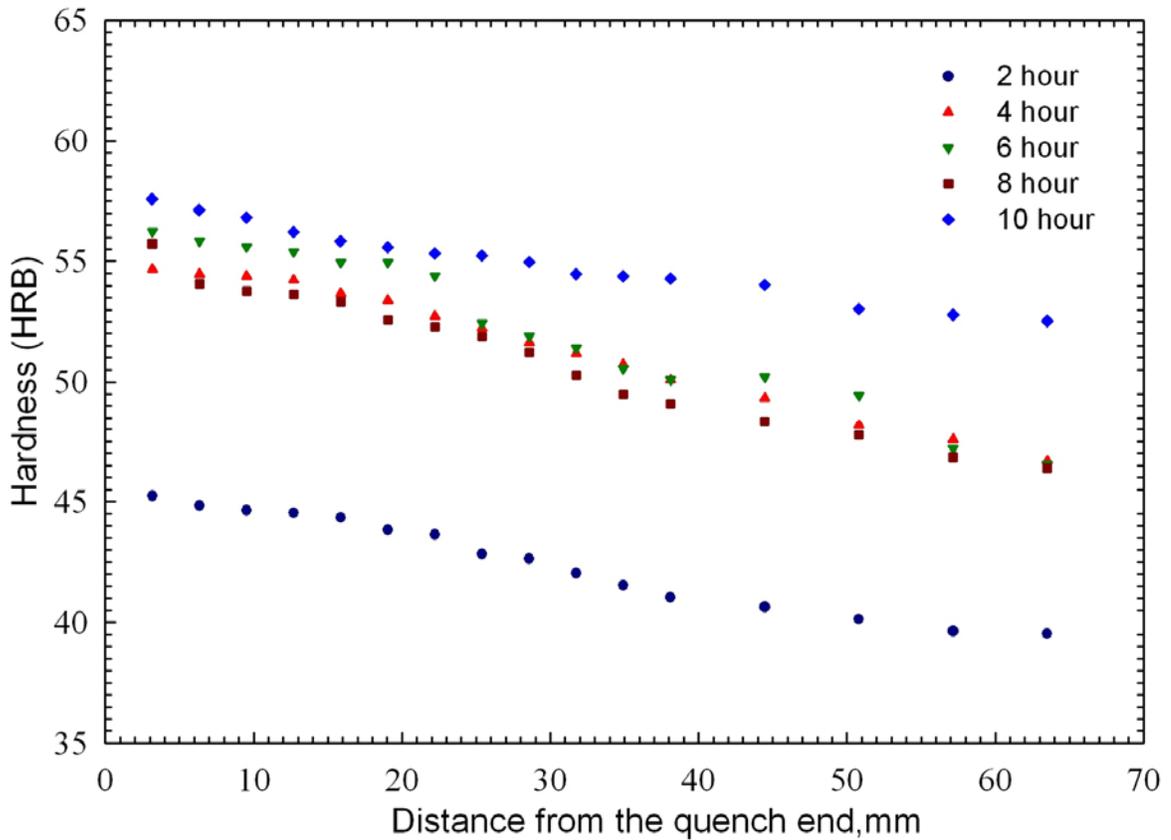


Figure 8 Hardness profile of a Jominy End Quench bar of cast aluminum alloy A356 with different aging times

Based on the experimental plan given in Table II, a series of experiments were performed to study the effect of aging time on the hardness of cast

aluminum alloy A356. The results are plotted in Figure 8. A gradual decrease in hardness is observed along Jominy End Quench bars, which results from the decrease in cooling rate during the quenching process. A two-hour aging time gives the lowest hardness, which is in the range of 40HRB. Aging times greater than 2 hours increase the hardness dramatically. The highest hardness is from the 10 hour aging. In the scope of this study, the over-aging phenomenon is not seen. Aging times of 2 hours in a conventional furnace will not result in an acceptable strength of cast aluminum alloy A356.

C. Quench Factor modeling

For Quench Factor modeling, both the thermal history of an alloy and the mechanical properties, which result from specific quenching rates, need to be obtained.

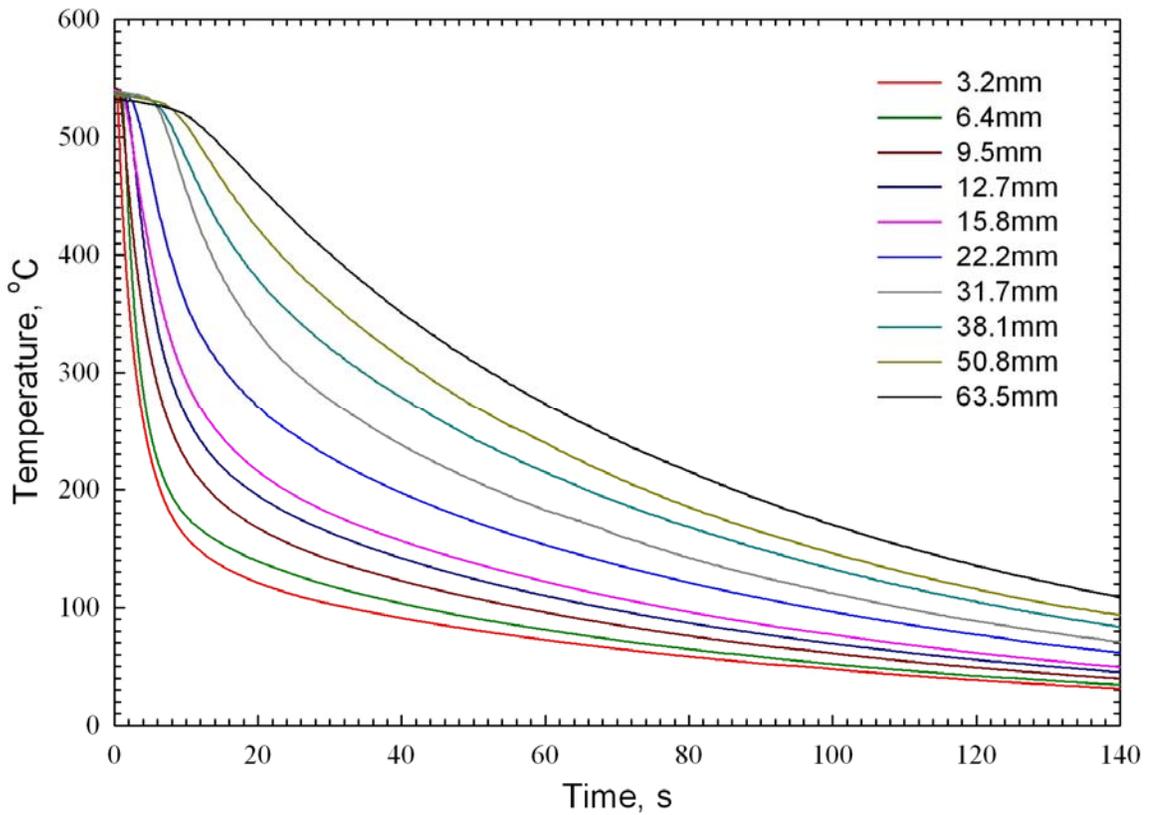
Table III. Distance from the quench end where experimental data was collected.

mm	3.2	6.4	9.5	12.7	15.8	22.2	31.7	38.1	50.8	63.5
Inch($\times 1/16$)	2	4	6	8	10	14	20	24	32	40

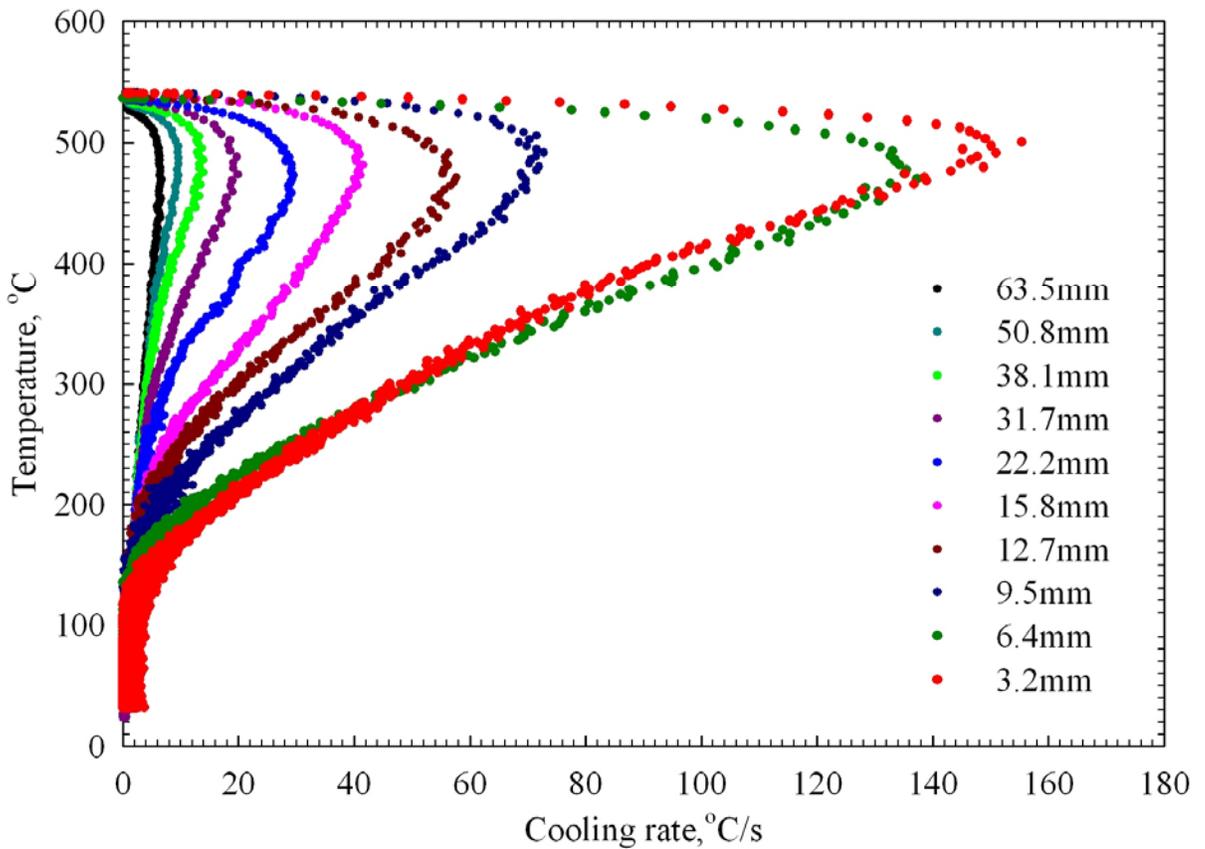
The thermal history of cast aluminum alloy A356 was obtained by measuring the time-temperature data with K-type thermocouples during quenching process at selected locations of a Jominy End Quench bar after the bar was solutionized at 540°C for 4 hours. The selected locations are given in Table III. The locations are selected to cover a wide range of cooling rates. The

temperature and cooling rate profiles at different locations of a Jominy End Quench bar are presented in Figure 9 (a) and (b).

Due to the nature of axial cooling along the bar, a large variation in cooling rate is observed. At the point of 3.2mm from the quench end, the maximum cooling rate is approximately 150°C/s , which is equivalent to water quench. The maximum cooling rate decreases dramatically to about 5°C/s at 63.5mm from the quench end, similar to the cooling rate attainable from an air quench. A large range of cooling rates, from the fastest to the slowest, can be attained using a Jominy End Quench bar.



(a) Temperature vs. time



(b) dT/dt vs. temperature

Figure 9 Cooling curves (a) and cooling rate curves (b) at different locations of a Jominy End Quench bar of cast aluminum alloy A356

The mechanical property used in this analysis is the Meyer hardness, which has an approximate linear relationship with the strength, so the assumptions for the Quench Factor analysis are valid in this case. Meyer hardness values were obtained from the conversion of Rockwell B hardness values with the relationship established by Tiryakioglu and Campbell [22]. Two flats, milled down 0.381mm from the surface, were machined from a Jominy End Quench bar aged for 6 hours at 165°C. Rockwell B hardness measurements were made

at the locations where the time-temperature data was collected. The Meyer hardness is plotted vs. distance from the quench end in Figure 10. The hardness value ranges from 143MPa at 3.2mm from the quench end to 130MPa at 63.5mm from the quench end.

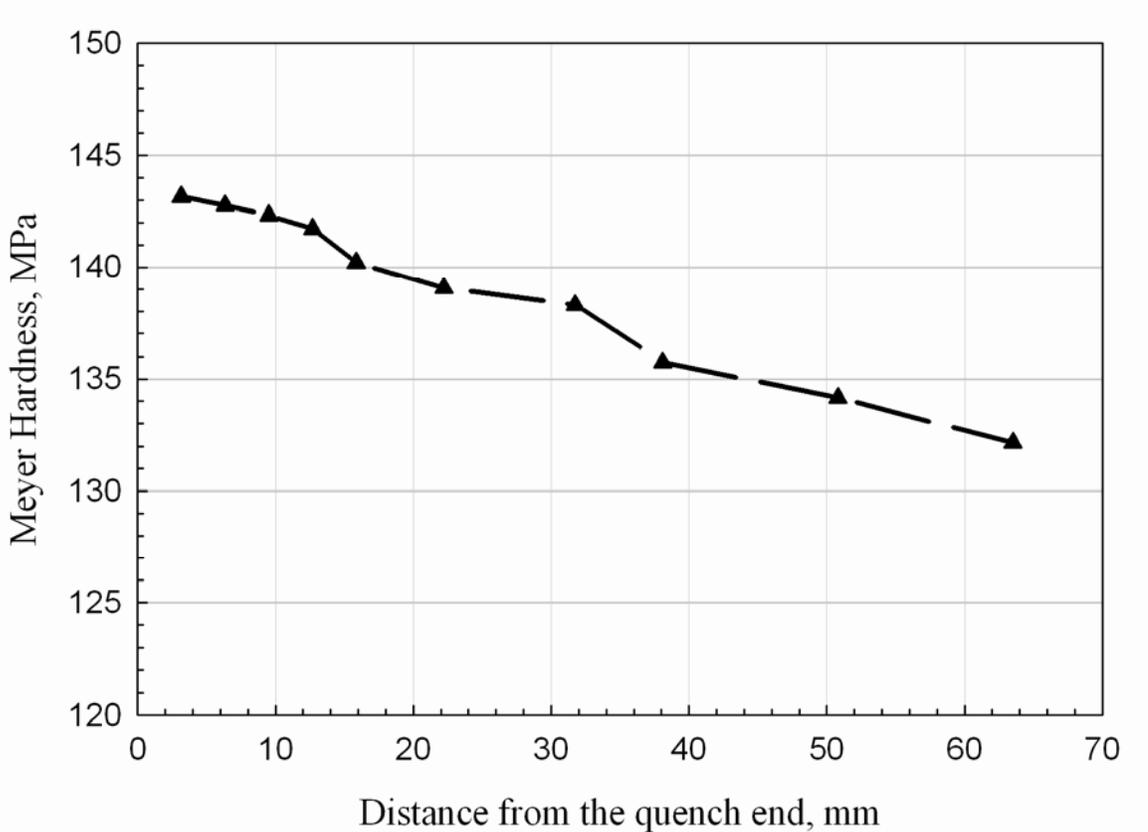


Figure 10 Meyer hardness along a Jominy End Quench bar of cast aluminum alloy A356

The maximum Meyer hardness, \bar{P}_{\max} , in Equation (3) is taken as the value at the quench end since the quench end is subject to the most severe cooling and only limited precipitation is assumed to possibly occur during quenching. To obtain the minimum Meyer hardness \bar{P}_{\min} in equation (3), a Jominy End Quench bar was solutionized at 540°C for 4 hours in a conventional furnace

and then transferred to a fluidized bed that was pre-heated to 540°C. The heater was turned off and the blower was left on. The test bar cooled slowly in the fluidized bed for about 20 hours to allow the precipitation to approach the equilibrium state [18]. The bar was then quenched in the water. The as-quenched sample was aged at 165°C for 6 hours in a conventional furnace. Hardness was measured on the cross section of the as-aged specimen. Ten readings were taken and averaged to obtain the minimum hardness used in the Quench Factor models.

Among the techniques available in the literature for determining the kinetic parameters, multiple linear regression analysis was employed in this paper. This technique was used by Rometsch to estimate the kinetic parameters for sand cast Al-7Si-Mg alloys in terms of yield strength [18]. Instead of minimizing the squares of the difference between the predicted and measured property as described in the least squares routine, this method is used to obtain a best linear relationship between a function of experimentally measured properties and the calculated Quench Factors.

If double natural logarithms are taken on both sides of Equation (3), then the following equation is generated. Since the relationship between strength and Quench Factor in Equation (3) is valid, the logarithm of fractional Meyer

hardness has a linear relationship with $\text{Ln}(Q)$ with the intercept being Avrami exponent n , as shown in Equation (7).

$$\text{Ln} \left[\frac{1}{K_1} \text{Ln} \left(\frac{\sigma - \sigma_{\min}}{\sigma_{\max} - \sigma_{\min}} \right) \right] = n \text{Ln}(Q) \quad (7)$$

The left side of the equation can be calculated with the known maximum, minimum hardness, and the measured hardness at the selected locations of a Jominy End Quench bar. Together with experimentally measured quenching data in Figure 9, K constants in Equation (1) are initially estimated to calculate the Quench Factors Q at the same locations using Equation (2). The logarithm of fractional Meyer hardness is plotted against $\text{Ln}(Q)$ as a scatter plot. The scatter plot is fitted with a linear curve and coefficient of determination (R^2) for the curve is calculated [18]. The constants in Equation (1) are iteratively adjusted until the hypothetical Quench Factors provide the highest possible coefficient of determination for the plot while the fitted linear curve passes through the origin (or the intercept is very close to 0) [18].

An example best-fit curve using Equation (3) is shown in Figure 11. The kinetic parameters and Avrami exponent obtained from multiple linear regression analysis are presented in Table IV. The constants for the improved model in Equation (4) are obtained by the same analysis and presented in Table IV.

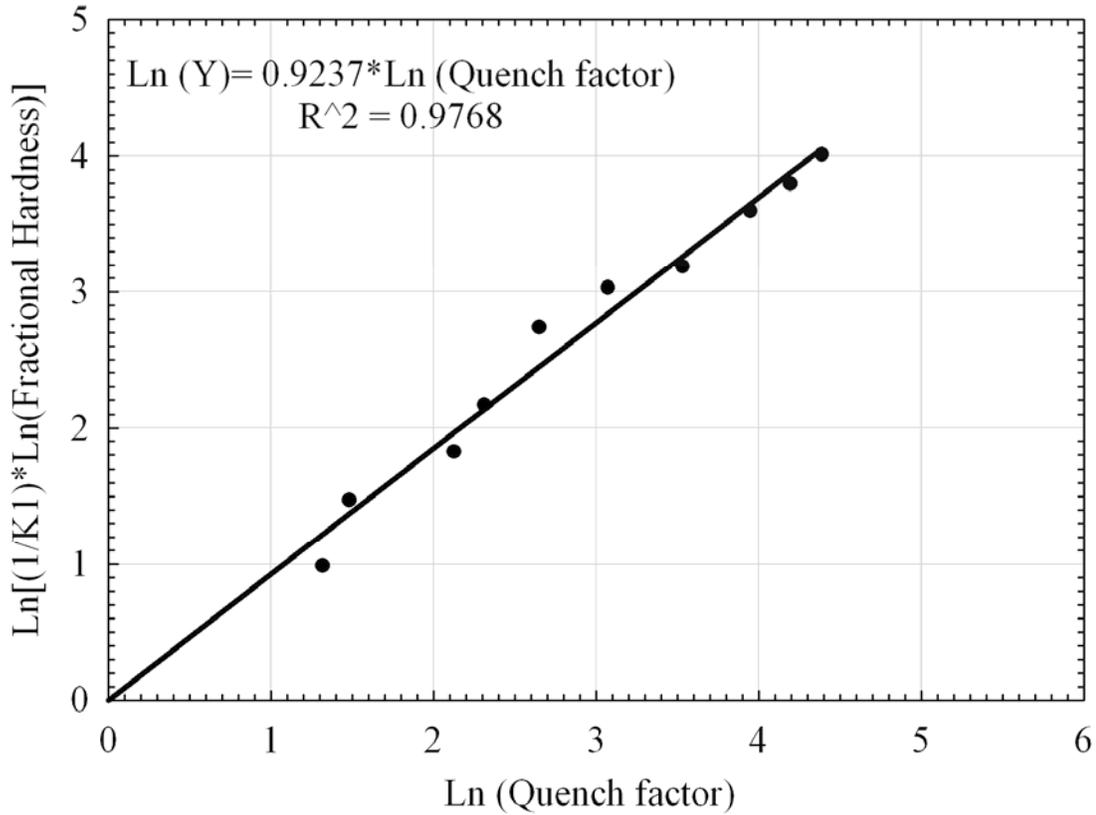


Figure 11 An example best fit curve for Quench Factor analysis of cast aluminum alloy A356 (0.5% precipitation)

Table IV. Precipitation kinetic parameters of cast aluminum alloy A356 during quenching process

	K_1	K_2	K_3 (J/mol)	K_4 (K)	K_5 (J/mol)	Avrami exponent, n
Equation (3)	-0.00513	1.27E-09	60	764	131000	0.92
Equation (4)	-0.00513	6.41E-10	56	764	131000	0.92

With the constants given in Table IV, the critical times were calculated using Equation (1) and plotted as a function of temperature for both original and improved Quench Factor models, as shown in Figure 12. These two curves correspond to 0.5% precipitation for cast aluminum alloy A356.

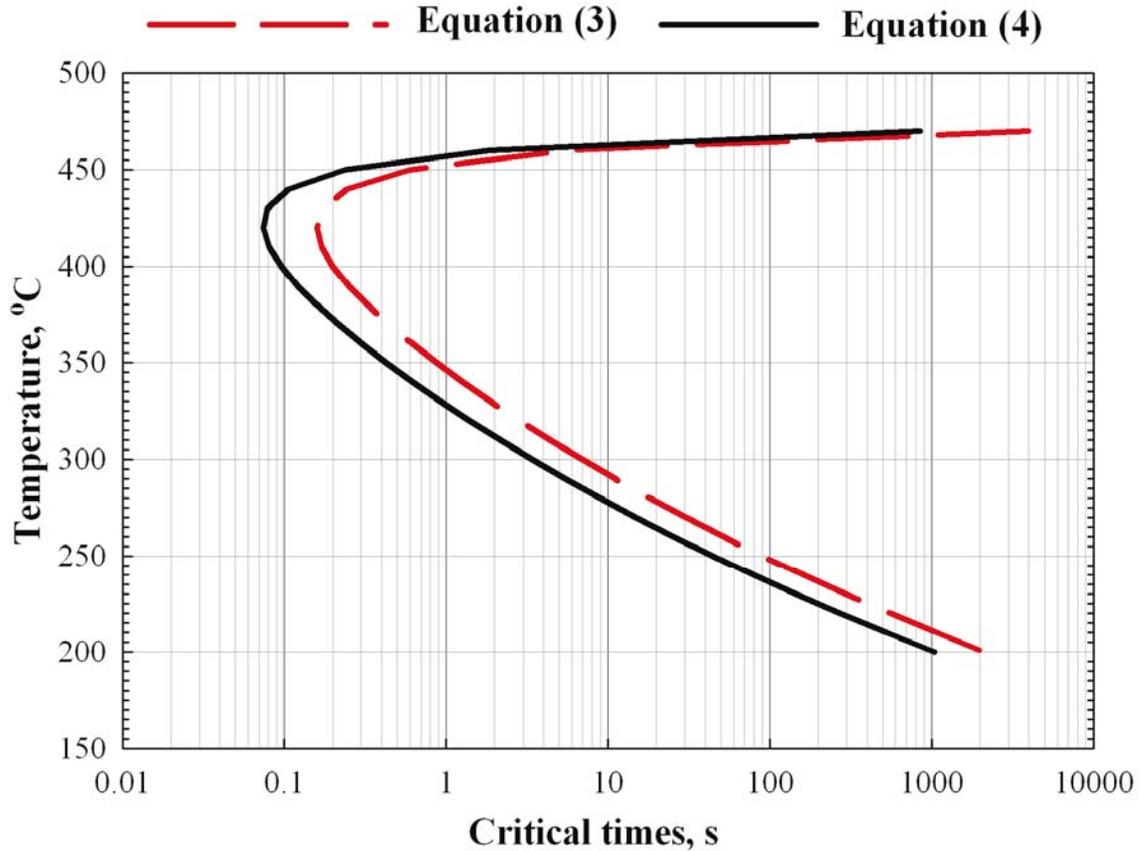


Figure 12 Time-Temperature-Property curves for cast aluminum alloy A356.

D. Experimental verification

Experimental verification was performed using five-cylinder cast aluminum A356 engine cylinder head, which was cast using the lost foam casting process. Sixty four engine heads were placed in a quench load, in 2 layers (2x32) in a continuous furnace, as shown in Figure 13 [19]. One of the engine heads was instrumented with K-type thermocouples to record the time-temperature data during the quenching process. One engine head was selected for the purpose of mechanical testing and metallographic

investigation. The rest of the engine cylinder heads were used as dummies to study the effect of racking pattern.

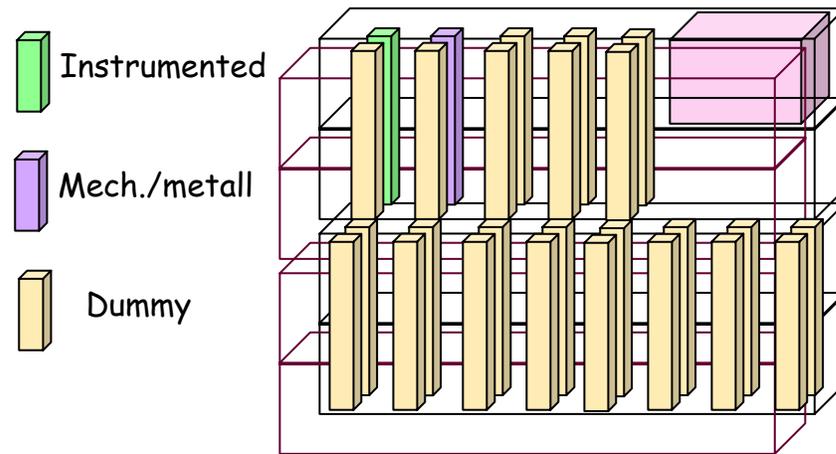


Figure 13 Racking pattern of cast aluminum A356 engine heads in a continuous furnace [19]

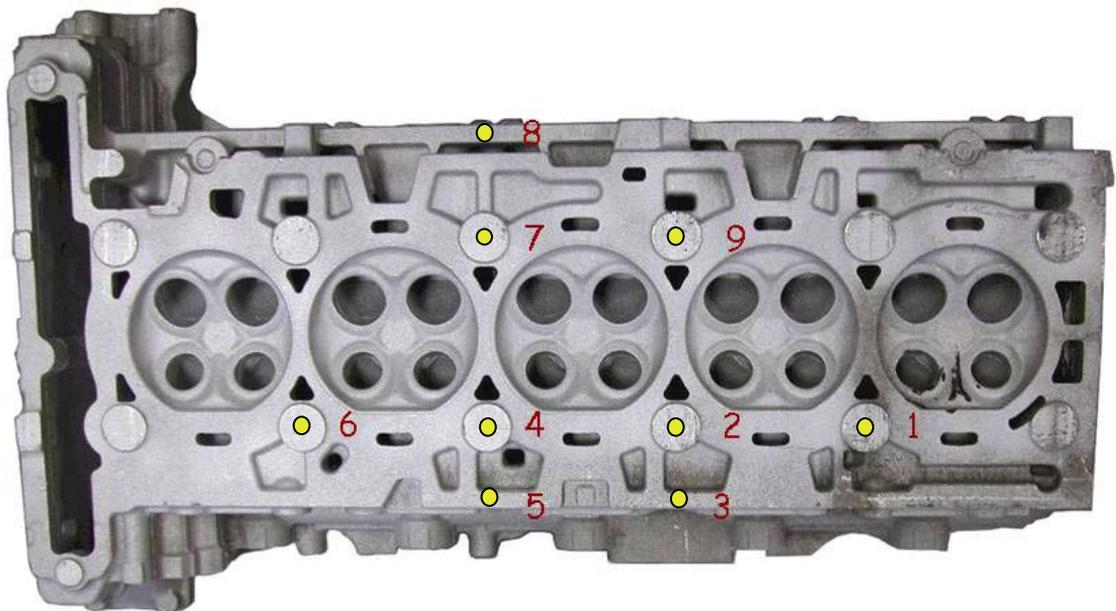


Figure 14 Cast aluminum A356 engine head instrumented with K-type thermocouples [19]

The engine heads were solutionized at 538°C (1000°F) for 5 hours in a continuous furnace (7 hours including the ramp-up time) and quenched in agitated water at 76°C (170°F). As shown in Figure 14, K-type thermocouples were instrumented at the selected 9 locations of one five-cylinder engine head and time-temperature data was collected at these locations during quenching process. As-quenched engine cylinder heads were aged at 160°C (320°F) for 4 hours (6 hours including the ramp-up time). As-aged samples were used for metallography and mechanical testing.

Table V. Predicted and measured hardness of a cast A356 engine head

	Location 7	Location 8
Measured hardness (HRB)	58.5 (± 0.8)	59.4 (± 1.0)
Predicted hardness (Equation (3))	59.0	59.5
Predicted hardness (Equation (4))	58.9	59.3

Two specimens were removed from the locations where thermocouples 7 and 8 were attached to the cast aluminum A356 engine head. Rockwell B hardness measurements were taken near the spot where the thermocouple tips were attached using a Wilson hardness tester Model 3JR, S/N 10661. The results are shown in Table V. Using the time-temperature data collected at the corresponding two locations, the Meyer hardness was predicted with the kinetic parameters given in Table IV and converted to Rockwell B hardness. The predicted hardness data was compared with the measured hardness,

with the results shown in Table IV. The predicted hardness agreed well with the measured one. These results have also been presented elsewhere [19].

VI. SUMMARY

The effects of solutionizing time, quenching rate and aging time on the microstructure and mechanical properties of age-hardenable cast aluminum alloy A356 were experimentally investigated with the Jominy End Quench approach. The results indicated that,

- The solutionizing time for permanent mold cast alloys could be reduced from 10 hours to 4 hours or less depending on the casting microstructure and secondary dendrite arm spacing.
- The aging time increased the hardness of cast aluminum alloy A356 in the range of 2 hours to 10 hours.
- With the experimentally measured quenching rates and Meyer hardness along a Jominy end quench bar, the kinetic parameters for cast aluminum alloy A356 were estimated using multiple linear regression analysis for Quench Factor modeling.
- Time-Temperature-Property (TTP) curves for cast aluminum alloy A356 were generated with the estimated kinetic parameters.
- Experimental verification was performed with a L5 engine head of cast aluminum alloy A356. The predicted property agreed well with that experimentally measured.

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Chapter IV Conclusions

In polymer quench, the concentration of aqueous polymer solution and the agitation are two important process parameters. In this study, the effects of process parameters, polymer concentration and agitation, on the quenching behavior of cast aluminum alloy A356 in aqueous solution of Aqua-Quench 260 were investigated using the CHTE quenching-agitation system. The test matrix was designed with the Taguchi technique and the experimental results were analyzed with analysis of variance (ANOVA) based on the average cooling rate. The average cooling rate dramatically decreased with the increase in polymer concentration. The agitation only enhanced the average cooling rate at low and medium levels. When high agitation was employed, average cooling rate dropped. From the results of ANOVA, the dominating process parameter in influencing the variation of average cooling rate was the polymer concentration; its percentage contribution was 97%. The effects from agitation and the interaction between polymer concentration and tank agitation were insignificant. Under all the heat treatment conditions, the micro-hardness increased with the aging time to a peak value and then decreased with a prolonged aging time. Water quenched sample showed the highest hardness. The increase in the polymer concentration lowered the attainable hardness for polymer quenched samples. Air cooled samples exhibited the lowest hardness as expected.

From energy savings point of view, research has been focusing on examining the possibility of shortening the heat treatment cycle, especially reducing the solutionizing and aging time without sacrificing the mechanical properties to a great extent. In this study, the effects of solutionizing times, quenching rates and aging times on the microstructure and mechanical properties of age-hardenable cast aluminum alloy A356 were experimentally investigated with the Jominy end quench approach. The results indicated that the solutionizing time for permanent mold cast alloys could be reduced from 10 hours to less than 4 hours depending on the casting microstructure and secondary dendrite arm spacing. The aging time increased the hardness of cast aluminum alloy A356 in the range of 2 hours to 10 hours. In the literature, quench factor analysis was proved to be an effective tool to quantify the reduction in strength from a slow quench. With the experimentally collected quenching rates and Meyer hardness along a Jominy end quench bar, the kinetic parameters for cast aluminum alloy A356 were determined using multiple linear regression analysis for Quench Factor modeling. Time-Temperature-Property (TTP) curves for cast aluminum alloy A356 were generated with the estimated kinetic parameters. Experimental verification was performed on a L5 engine head of cast aluminum alloy A356. The predicted property agreed well with that experimentally measured.