

**DEVELOPMENT OF A MATHEMATICAL MODEL  
TO RELATE THE AGEING PARAMETERS TO  
HARDNESS AND TENSILE STRENGTH OF AL  
6063 ALLOY**

Selvarathinam Sivanujan

218010G

Master of Science (Major Component of Research)

Department of Materials Science and Engineering  
Faculty of Engineering

University of Moratuwa  
Sri Lanka

August 2023



**DEVELOPMENT OF A MATHEMATICAL MODEL  
TO RELATE THE AGEING PARAMETERS TO  
HARDNESS AND TENSILE STRENGTH OF AL  
6063 ALLOY**

Selvarathinam Sivanujan

218010G

Thesis submitted in partial fulfillment of the requirements for the degree  
Master of Science (Major Component of Research)

Department of Materials Science and Engineering  
Faculty of Engineering

University of Moratuwa  
Sri Lanka

August 2023

## DECLARATION

I declare that this is my own work and this Thesis does not incorporate without acknowledgement any material previously submitted for a Degree or Diploma in any other University or Institute of higher learning and to the best of my knowledge and belief it does not contain any material previously published or written by another person except where the acknowledgement is made in the text. I retain the right to use this content in whole or part in future works (such as articles or books).

Signature: *UOM Verified Signature*

Date: 30/08/2023

The above candidate has carried out research for the Master of Science (Major Component of Research) Thesis under my supervision. I confirm that the declaration made above by the student is true and correct.

Name of Supervisor: Dr. GIP De Silva

Signature of the Supervisor:

Date: 30/08/2023

*UOM Verified Signature*



## **ACKNOWLEDGEMENT**

This work is not achieved only by myself, there are support of individuals and groups, even though a word was a strength towards full filling my research objectives. I wish to acknowledge all of them who helped me in completing this work successfully. First and foremost, I would like to express my heartiest gratitude with respect to my supervisor Dr. G.I.P. De Silva. He provided me with a great opportunity to join with this research project and has given all the direction, encouragement and guidance to achieve the goal throughout the entire period to accomplish my objectives. His constructive feedback and advice from the start to end improved my research ability and professional skills.

I would like to express my deepest gratitude to Dr. (Mrs.) S.C. Mathugama from Institute of Technology University of Moratuwa for her guidance and support in the objective of development of mathematical model. Also, I would like to thank Prof. M. Narayana and Prof. A.S. Galhenage for their immense support and academic encouragements throughout the evaluation procedure. Next, I would like to thank SRC grant committee for the funding assistance and Alumex for providing Al 6063 samples for research works.

Further, I would like to thank Mr. V. Sivahar/ Head of Department, all academic and nonacademic staff of department of materials science and engineering, University of Moratuwa for supporting me to conduct my research works in a better academic environment. Also, I would like to thank all of my fellow research assistants who were always there with me providing me a friendly and smooth platform to carry out this research project in a successful manner. Lastly, I convey my love and gratitude to my parents for their constant support in the every moment of my life.



## ABSTRACT

Deformations, failures, and the wearing-off effect are common in Al 6063 structures due to their low strength and hardness, respectively. Industries have age-hardened Al 6063 alloy to improve its properties to a specified level depending on the components being produced. Industries do trials before production and check to see if the product has achieved its required levels of properties. This trial-and-error method is time-consuming, and further, it is not acceptable from an engineering perspective. For this reason, industries are looking for a model that will provide an accurate prediction of the hardness and tensile strength for the parameters associated with aging.

In this research, a mathematical model was developed to predict the most efficient combination of aging parameters to achieve the required tensile strength and hardness of Al 6063. The model was developed based on the experimental tensile strength and hardness values for the 25 combinations obtained by varying aging time and temperature at five levels. Tensile strength and hardness were measured using the universal tensile testing machine and the Vickers hardness tester, respectively. Further, the model was developed using the SPSS statistical software and validated with data sets obtained from the literature. For the purpose of finding the most efficient combination of tensile strength and hardness, the model was developed as a computer program based on the Python programming language.

In addition to the development of the model, the influence of precipitate size distribution on the tensile strength and hardness variation of Al 6063 alloy with aging temperature and time was investigated. Micro-structures were observed, and precipitate types were identified using a scanning electron microscope and an energy dispersive spectrometer (SEM/EDS). The precipitate size distribution was determined based on SEM images using MIPAR image analysis software. Beyond the peak age stage, a significant increase in the percentage of precipitates larger than 1.5  $\mu\text{m}$  and a decrease in the percentage of precipitates smaller than 0.75  $\mu\text{m}$  were accompanied by a decrease in tensile strength and hardness.

**Keywords:** aluminum 6063, age hardening, hardness, tensile strength, precipitate size distribution, mathematical modeling





## TABLE OF CONTENTS

Declaration of the Candidate & Supervisor	i
Acknowledgement	iii
Abstract	v
Table of Contents	vii
List of Figures	xi
List of Tables	xv
List of Abbreviations	xv
List of Appendices	xix
1 Introduction	1
1.1 Research problem	2
1.2 Research Background	2
1.3 Significance of Research	3
1.4 Objectives	3
1.5 Scope of the Thesis	4
2 Literature Survey	5
2.1 Background of age hardening	5
2.2 Homogenisation during the production	6
2.3 Age hardening	7
2.3.1 Solution treatment	7
2.3.2 Quenching	8
2.3.3 Aging treatment	9
2.3.4 Etching methods	10
2.3.5 Effect of other treatment before the aging	11
2.3.6 Precipitation during aging	11
2.3.7 Strengthening during aging	12
2.4 Effect of aging parameters on micro-structure and mechanical properties	15
2.5 Previous models related to age hardening	17

2.6	Statistical methods for data analysis	20
2.7	Summary of literature survey	21
3	Methodology	23
3.1	Chemical Analysis	23
3.2	Sample Preparation	24
3.3	Experimental Trail	25
3.4	Heat Treatment – Solution Treatment, Quenching and Aging	27
3.5	Measuring hardness and tensile strength for each sample	28
3.6	Micro-structure Analysis with SEM/EDS	29
3.7	Precipitate Size Distribution Analysis with MIPAR Software	31
3.8	Mathematical Model: Aging Parameters and Mechanical Properties	32
4	Results and Discussion	35
4.1	Experimental trail results	35
4.2	Variation of mechanical properties with aging parameters	37
4.3	Microstructural analysis and identification of precipitates	39
4.4	Influence of precipitate size distribution on the variation of mechanical properties	42
4.4.1	Percentages of precipitates within specific ranges	42
4.4.2	Mean precipitate size within specific ranges	45
4.5	Development of a Mathematical Model	46
4.5.1	Tests of assumptions	46
4.5.2	One-factor ANOVA	48
4.5.3	Two-factor ANOVA without and with interaction	49
4.5.4	Correlation between hardness and tensile strength	50
4.5.5	MANOVA, including the interaction effect	51
4.5.6	Model 1: A mixed model for strength and hardness	54
4.5.7	Model 2: A model by regression equation	55
4.6	Validation of mathematical model	57
4.6.1	Validation of model based on the predictions for aging parameters	57
4.6.2	Validation of model based on literature results	58

4.7	Implementation and limitations of the model	59
4.7.1	Implement the model for the application	59
4.7.2	Limitations during the application	60
5	Conclusions and Recommendations	61
	References	63
	Appendix A Drawing of specimen used for the tensile testing	71
	Appendix B The variation of Precipitate Size Distribution and Mechanical Properties against Aging Parameters	73
	Appendix C Results of One factor ANOVA	75
	Appendix D Results of two factor ANOVA	77
	Appendix E The results of “Tests of Between-Subjects Effects” from ‘Two-way MANOVA’	79
	Appendix F Results of fixed effects from mixed model	81



## LIST OF FIGURES

Figure	Description	Page
Figure 1.1	Significance of research	3
Figure 2.1	A pseudo-binary diagram of Al-Mg <sub>2</sub> Si	8
Figure 2.2	Hardness after artificially aged against cooling rate	9
Figure 2.3	Image of equilibrium structure to solid solution to precipitates	10
Figure 2.4	HRTEM images of cryorolled Al 6063 after the aging	12
Figure 2.5	Net Aging Strength Curve	13
Figure 2.6	Dislocation cutting through the particle	14
Figure 2.7	Dislocation interacts with widely spaced particles	14
Figure 2.8	(a) A systematic graphic illustrating dislocation movement by shearing or bypassing; (b) A correlation between the radius of the precipitate and its strength to resist shearing or bypassing of dislocations	15
Figure 2.9	DSC plot of solution treated Al 6063 alloy samples at a heating rate of 20 °C/min	16
Figure 2.10	Comparison of the Deschamps and Brechet's model predicted (a) precipitate radius with Small Angle Scattering and TEM data, (b) yield stress with micro-hardness data	18
Figure 2.11	Comparison between observed and Myhr et al.'s model predicted (a) mean particle radius, (b) hardness of Al 6005 alloy	18
Figure 2.12	Prediction of yield strength by AMAP model for different aging temperatures and alloy composition	20
Figure 3.1	Flow chart of the methodology	23
Figure 3.2	Initial cutting of disc shape with thickness of 14 mm and 10 mm from the billet	24
Figure 3.3	VERTEX Precision Machine Tool's 7" cutting band saw	25
Figure 3.4	Dumbbell-shaped sample used for tensile testing	25
Figure 3.5	Heat treatment process of Treatment 4	26
Figure 3.6	Heat Treatment Process; T – Temperature for Solution Treatment (540 °C), T <sub>x</sub> – Aging Temperature to be varied, t <sub>s</sub> – Holding Time for Solution Treatment (90 mins) and t <sub>x</sub> – aging time to be varied	27
Figure 3.7	A furnace used during Solution treatment	27
Figure 3.8	ISOMET Slow Speed Saw	29
Figure 3.9	OSK Vickers Hardness Testing Machine	29
Figure 3.10	Measuring hardness	30

Figure 3.11	User interface of universal testing machine with metallic material— tensile testing while testing the specimen of combination 23 (Aging Temperature 210 °C and Aging Time 360 minutes)	30
Figure 3.12	Stress–Strain Curve of Combination 23 (Aging Temperature 210 °C and Aging Time 360 minutes)	31
Figure 3.13	A graphical representation of the equivalent and minimum diameters	32
Figure 3.14	Analytical methods	32
Figure 3.15	Interface of SPSS software with input data	33
Figure 3.16	Interface of Minitab software	33
Figure 4.1	SEM images with precipitate for the Experimental Trial	36
Figure 4.2	SEM images with etchants; (a) Etchant 1: A solution containing 25 ml of methanol, 25 ml of HNO <sub>3</sub> , 25 ml of HCl and one drop of HF (b) Etchant 2: Keller’s solution (1.0 ml HF, 1.5 ml HCl, 2.5 ml HNO <sub>3</sub> , and 95.0 ml distilled water)	36
Figure 4.3	EDS Spot analysis on side by side Precipitates	37
Figure 4.4	(a) EDS Spot Analysis Inside and Outside of Precipitate and (b) EDS Line Analysis through precipitate and matrix	37
Figure 4.5	Tensile strength against aging temperatures for different aging times	38
Figure 4.6	Hardness against aging temperatures for different aging times	39
Figure 4.7	SEM images for the aging times of (a) 225 minutes (before peak age), (b) 360 minutes (after peak age) and (c) 315 minutes (peak age) at 210°C	40
Figure 4.8	SEM images of aging at (a) 190 (Near Peak Age) and (b) 230 (Peak Age) for 315 minutes	41
Figure 4.9	SEM/EDS spot analysis (a) inside and (b) outside of formed precipitate for the aging combination of (170°C, 270 minutes)	42
Figure 4.10	SEM/EDS spot analysis (a) inside and (b) outside of formed precipitate for the aging combination of (210°C, 270 minutes)	42
Figure 4.11	Precipitate size percentages within specific ranges for different aging temperatures for the aging times of (a) 180 min, (b) 225 min, (c) 270 min, (d) 315 min, and (e) 360 min)	43
Figure 4.12	The results from boxplots	46
Figure 4.13	Test of normality with Shapiro-Wilk	47
Figure 4.14	Box’s M Test: aging (a) temperature and (b) time	47
Figure 4.15	Scatter plots for strength and hardness by aging (a) time and (b) temperature	47
Figure 4.16	Collinearity statistics of (a) Hardness and (b) Tensile strength	48
Figure 4.17	The results of one-factor ANOVA for the effect of aging temperature on tensile strength.	49
Figure 4.18	The results of one-factor ANOVA for the effect of aging time on tensile strength.	49

Figure 4.19	Ageing time, temperature, and the effect of their interaction on (a) tensile strength and (b) hardness were studied using a two-factor ANOVA	50
Figure 4.20	Correlation between “hardness” and “tensile strength”	51
Figure 4.21	The results of “Multivariate Tests” from ‘Two-way MANOVA’	51
Figure 4.22	Scatter plots for the model: Intercept + Aging Temperature + Aging Time + Aging Temperature * Aging Time	53
Figure 4.23	Analysis of variance for the regression equation of (a) hardness and (b) tensile strength	56
Figure 4.24	Comparing the actual values from experimental and predicted values from the mixed model (1) and categorical predictor model (2) for (a) tensile strength and (b) hardness	57
Figure 4.25	Comparing the predicted values from the both Models with the data sets obtained from the literature	59
Figure 4.26	Finding the best combination with Python based model	60





## LIST OF TABLES

Table	Description	Page
Table 1.1	Aluminum alloy series, their major alloying elements and hardenability	1
Table 2.1	Summary of solution treatment and quenching prior to age hardening used by previous researchers.	7
Table 3.1	Chemical composition of Al 6063 alloy obtained from the local industry and in accordance with standard	23
Table 3.2	Varying combinations of aging parameters	28
Table 3.3	Assumption for ANOVA and MANOVA and their testing methods	34
Table 4.1	Results obtained during the experimental trial	35
Table 4.2	Percentages of precipitates based on sizes for aging times of 225, 315, and 360 min at 210 °C	39
Table 4.3	Element weight ratio of Fe- Si- rich precipitate	41
Table 4.4	Element weight ratio of Fe- Si- Mg- rich precipitate	41
Table 4.5	Mean precipitate size (MPS) and percentage of precipitates (PP) within R1 (< 0.75 $\mu\text{m}$ ), R2 (0.75 to 1.5 $\mu\text{m}$ ), and R3 (> 1.5 $\mu\text{m}$ )	45
Table 4.6	Summary of results based on Two-way MANOVA's "Tests of Between-Subjects Effects"	52
Table 4.7	Coefficients for combination (170 °C, 180 minutes) from 'parameter estimates' of two-way MANOVA	53
Table 4.8	Coefficients for combination (190 °C, 180 minutes) from 'parameter estimates' of two-way MANOVA	54
Table 4.9	Coefficients of 2 <sup>nd</sup> order polynomial equations	54
Table 4.10	Regression equation terms and their coefficients for hardness and tensile strength	55
Table 4.11	Aging combinations given in numbers in Figure 4.25 and data sets	58



## LIST OF ABBREVIATIONS

<b>Abbreviation</b>	<b>Description</b>
ANOVA	Analysis of Variance
BSD	Backscattered Electron Detectors
DSC	Differential Scanning Calorimetry
EDS	Energy Dispersive Spectroscopy
FEA	Finite Element Analysis
NN	Neural Network
RSM	Response Surface Methodology
SEM	Scanning Electron Microscopy



## LIST OF APPENDICES

<b>Appendix</b>	<b>Description</b>	<b>Page</b>
Appendix -A	Drawing of specimen used for the tensile testing	71
Appendix -B	The variation of Precipitate Size Distribution and Mechanical Properties against Aging Parameters	73
Appendix -C	Results of One factor ANOVA	75
Appendix -D	Results of two factor ANOVA	77
Appendix -E	The results of “Tests of Between-Subjects Effects” from ‘Two-way MANOVA’	79
Appendix -F	Results of fixed effects from mixed model	81