

Regresyon Analizi Kullanılarak A356 Alaşımında Stronsiyum İçeriği ve Soğutma Hızı Arasındaki İstatistiksel İlişki

Statistical Relationship Between Strontium Content and Cooling Rate on A356 Alloy by Using Regression Analysis

Batuhan Doğdu, Onur Ertuğrul
Malzeme Mühendisliği Bölümü, İzmir Katip Çelebi Üniversitesi, İzmir, Türkiye
dogdubatuhan@gmail.com, onur.ertugrul@ikcu.edu.tr

Özetçe—Ötektik silikon modifikasyonu, Al-Si alaşımları üzerinde, mekanik kabiliyet ve döküm parçanın enerji absorpsiyonu açısından önemli bir döküm parametresidir. Ticari olarak Al-Si alaşımı üzerinde ötektik modifikasyon elde etmek için kimyasal modifiye edici element stronsiyum kullanılmıştır. Öte yandan, Al-Si alaşımlarında yüksek soğutma hızı, hem dendritleri rafine eder hem de silikon fazı ile mekanik karakteristiğini artırır. Stronsiyum miktarı ile soğutma hızı arasında istatistiksel bir ilişki bulmak için, aynı döküm parçada farklı soğutma hızları aralığı elde etmek amacıyla bir kalıp tasarlanmış, ardından A356 alaşımının çekme testi verileri Minitab yazılımında analiz edilmiştir. Bu nedenle, regresyon ve varyans analizlerine geçildikten sonra, stronsiyum miktarının sadece $<0.9^{\circ}\text{C}$ olan düşük soğutma hızlarında baskın olduğu görülmüştür.

Anahtar Kelimeler— A356; ötektik modifikasyon; soğuma hızı; regresyon; ANOVA.

Abstract— Eutectic silicon modification is an important casting parameter on Al-Si alloys on the aspect of mechanical capability and energy absorption of the cast part. Chemical modifier element strontium has been used to obtain eutectic modification on Al-Si alloy commercially. On the other hand, high cooling rate on Al-Si alloys both refine dendrites and silicon phase which enhances mechanical characteristic. In order to find a statistical relationship between strontium amount and cooling rate, a special mold was designed in order to obtain different range of cooling rates in same cast part, then tensile test data of A356 alloy were analyzed in Minitab software. Therefore, after regression and analysis of variance tests have been proceeded, it was found that strontium amount is only dominant for lower cooling rates of $<0.9^{\circ}\text{C}$.

Keywords— A356; eutectic modification; cooling rate; regression; ANOVA.

I. INTRODUCTION

Aluminum (Al)-Silicon (Si) casting alloys as one of the most popular family of the cast Al alloys which have been used in industry for decades, especially for the advantages of their light weight, recyclability and high specific strength [1]. The most common composition of Al-Si casting alloy is A356 (Al7Si0.3Mg) which contains needle-like hard and brittle eutectic Si phase and ductile α -Al phase as alloy matrix. This eutectic phase, inside solidified commercially pure (CP) A356 alloy, has a needle-like structure and results an increment on inner stress due to increased stress sharp edges inside microstructure. Modification is a must in order to refine the eutectic Si phase to obtain finer morphology. Unmodified Si phase inside Al-Si casting alloy degrades tensile strength and fatigue properties of cast part In order to eliminate this negative effect and increase mechanical properties such as tensile strength, toughness and elongation, eutectic modification and grain refinement procedures have been used for most of the Al cast parts. This modification can be obtained by either chemical modification or fast heat extraction from molten alloy. Chemical modifiers are master alloys including strontium (Sr), sodium (Na), barium (Ba), calcium (Ca) and europium (Eu) elements used to obtain eutectic modification which avoids eutectic Si growth by the mechanism of impurity induced twinning (IIT) growth and twin plane re-entrant (TPRE) poisoning [2-4]. Besides, a high cooling rate between liquidus and solidus temperature makes for refining eutectic Si phase [5].

Statistical investigations on Al-Si casting alloys have made specifically on the aspect of fatigue life and tensile properties. Fatigue life performance of A356.2-T6 were studied by Ramamurty et. al statistically by using analysis of variance (ANOVA) [6]. On the other hand, pore size effect on fatigue performance of A356 alloy which

distinguished by Murakami's statistical method, were researched by Fintová et. al. [7]. Alexopoulos et al. examined the statistical distributions of effect of Samarium (Sm), silver (Ag), Si and Sr particle size and aspect ratio, as well as tensile properties. On the Si modification aspect, authors proposed that modification by Sr in A357-Cu alloy resulted increasing elongation due to Sr limiting the straightening of bifilms on higher Weibull distribution; conversely resulted major old oxides, on lower distribution [8]. Green and Campbell [9] also proposed the statistical relations between molten metal filling characteristic into the mold and fracture strength of A356 alloy.

However, even if statistical researches have been made by taking into account of eutectic Si modifier Al-Sr master alloy and cooling rate, by the outcomes of tensile properties such as yield strength, tensile strength, elongation and quality index (QI), statistical relationship between Sr addition amount and cooling rate are still an unknown phenomenon on Al-Si alloys [10].

$$QI = TS + 150\log(\%E) \quad (1)$$

The innovative objective of this study is to investigate the effect of Sr content and cooling rate on Si modification performance with the help of tensile test outputs.

II. MATERIALS AND METHODS

Firstly, a mold design was studied on Catia V5 CAD software by considering the cooling rate differences on each spoke. The five spoke mold was designed on two different section volume by referencing of middle point of the casting part. That section differences were designed to obtain different thermal gradient on the same mold. Therefore, on the thick section part, two air cooling nozzle holes were implemented to get much higher cooling rates due to get different cooling rates on edge to edge (higher values on thick section, lower values on thin section). After the design step, the CAD mold was appended on Magmasoft casting simulation software in order to observe solidification behavior of molten alloy in the mold. Cooling rate of each spoke were examined by a virtual thermocouple that were located on mid-point of each spoke (Fig. 1).

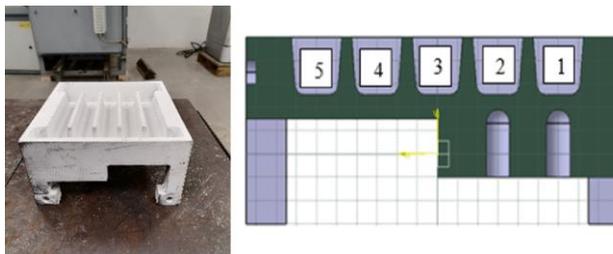


Figure 1. Isometric view, and mid-section of the mold, where cooling channel hubs and spoke numbers can be seen.

Five graphite crucibles were sintered for melting A356 ingot pieces. For each casting process, ingot pieces were weighted, as 2 kg for each five crucibles, then held in

electrical furnace in order to melt A356 ingots at 750 °C. After ingots were melted, Al-Sr master alloy additions were done in order to obtain required ppm levels which were 150, 250, 350 and 450 ppm Sr in A356 alloy. It was aimed to stabilize the molten A356 temperature and mold temperature for the purpose of obtaining simulation conditions, which were set as 700 °C as molten metal temperature and 350 °C as mold temperature. Castings were done into the mold. Cooling channels were switched to open on 6 bar dry air, after mold cavity were filled by molten metal (Fig. 2). In order to satisfy statistical consistency, 5 different compositions (CP A356, 150 ppm Sr, 250 ppm Sr, 350 ppm Sr and 450 ppm Sr addition) were cast 3 times with stabilized parameters (Table 1). All specimens were held to T6 heat treatment (Table 2). Cast part's chemical composition were examined with commercial optical emission spectrometer. Each spoke of cast part were analyzed 3 times, in order to data reliability of % Sr.

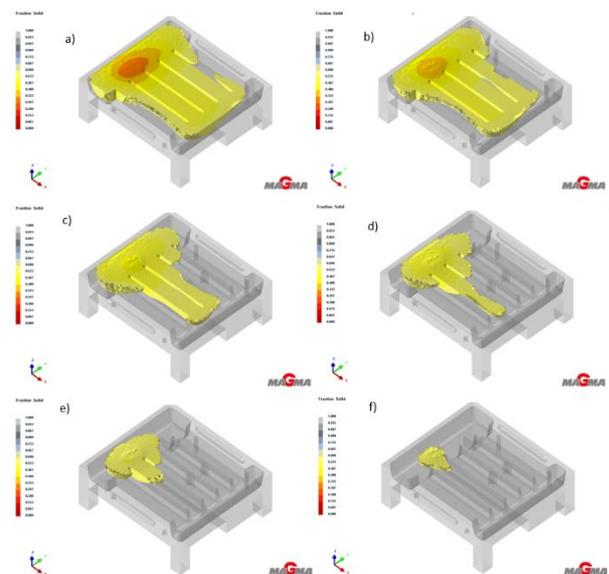


Figure 2. Solidification sequence of A356 Alloy at a) 65s, b) 75s, c) 95s, d) 115s, e) 135s, f) 175s as a result of % fraction solid.

Tensile test samples were prepared in accordance with DIN EN ISO 6892-1. Tensile tests were performed by Zwick Z100 Model tensile test machine with DIN EN 10002-1 test standard (as pre-load 5N/mm², test speed: 4.2mm/min.) Statistical analysis of relation between Sr amount and cooling rate were done on Minitab 18 software.

III. RESULTS AND DISCUSSION

In order to find the relationship between Sr amount and cooling rate, statistical characteristic of these data were performed on Minitab 18 by the outputs of Yield Strength and QI. Elongation and tensile strength data were eliminated due to being function parameters of QI. That elimination was ensured much more satisfied result on statistical comparison.

Fig. 3 shows all tensile test outputs. By performed regression analysis on Minitab 18, statistical relationship between Sr contribution rate and cooling rate were determined. Whole T6-heat treated tensile specimen data

put in regression analysis. This analysis is ensured us to obtain a model that relates continuous and categorical predictors with one response. In this analysis, YS and QI were selected as response, and $\alpha = 0.95$ significance level.

Table 1. Compositions of experimental setup.

Composition (%)	Al	Si	Mg	Fe	Cu	Ti	Sr
CP A356	92.4537	7.0343	0.2931	0.0993	0.0011	0.0877	0.0070
150 ppm Sr	92.0176	7.4664	0.2753	0.1104	0.0012	0.0894	0.0153
250 ppm Sr	92.4161	7.0436	0.2867	0.1057	0.0011	0.0870	0.0252
350 ppm Sr	92.1882	7.2261	0.3115	0.1285	0.0012	0.0857	0.0320
450 ppm Sr	92.1508	7.2658	0.3074	0.1061	0.0011	0.0883	0.0462

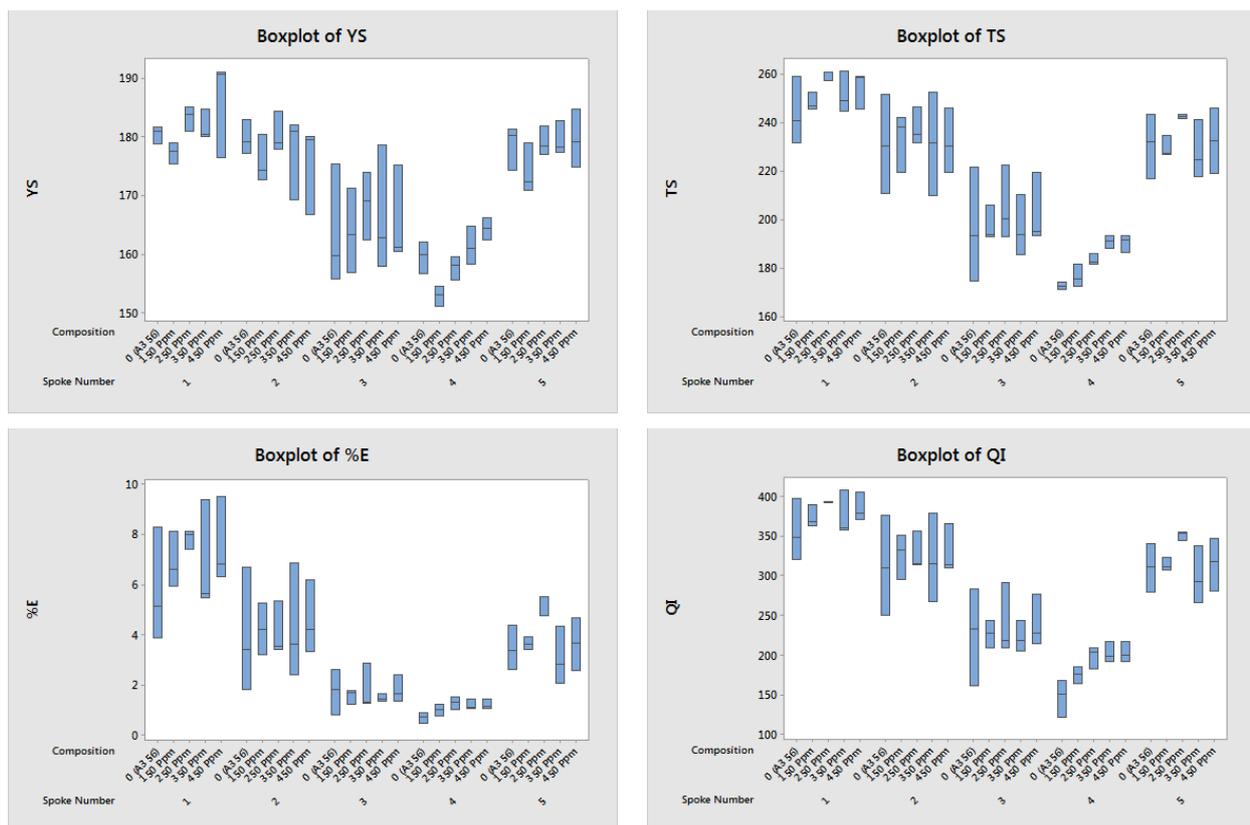


Figure 3. Tensile test result data of each sample.

R-square values obtained as 61.50% for YS and 73.89% for QI results (Fig. 4). It has been already known that higher the R-square value, more reliable the regression model. However, even if p-value is <0.05 for cooling rate, obtained Sr p-value is >0.05 . It was seen that this regression model does not have ability to propose relation between cooling rate and Sr addition. Cooling rate dominantly effected the regression, while Sr content could not explain the its effect statistically.

Table 2. Cooling rate of each spoke number.

Spoke Number	1	2	3	4	5
Cooling Rate ($^{\circ}\text{C}/\text{s}$)	2.1	1.4	1.2	0.9	1.7

Regression Analysis: YS versus Cooling Rate; Obtained Sr

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	2	4603,82	2301,91	57,51	0,000
Cooling Rate	1	4531,33	4531,33	113,21	0,000
Obtained Sr	1	91,05	91,05	2,27	0,136
Error	72	2881,89	40,03		
Lack-of-Fit	47	2223,25	47,30	1,80	0,059
Pure Error	25	658,64	26,35		
Total	74	7485,71			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
6,32663	61,50%	60,43%	58,58%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	142,24	3,12	45,52	0,000	
Cooling Rate	18,78	1,77	10,64	0,000	1,00
Obtained Sr	93,7	62,1	1,51	0,136	1,00

Regression Equation

$$YS = 142,24 + 18,78 \text{ Cooling Rate} + 93,7 \text{ Obtained Sr}$$

Regression Analysis: QI versus Cooling Rate; Obtained Sr

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	2	311220	155610	101,89	0,000
Cooling Rate	1	309029	309029	202,34	0,000
Obtained Sr	1	3058	3058	2,00	0,161
Error	72	109966	1527		
Lack-of-Fit	47	74815	1592	1,13	0,377
Pure Error	25	35150	1406		
Total	74	421185			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
39,0807	73,89%	73,17%	72,02%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	42,4	19,3	2,20	0,031	
Cooling Rate	155,1	10,9	14,22	0,000	1,00
Obtained Sr	543	384	1,41	0,161	1,00

Regression Equation

$$QI = 42,4 + 155,1 \text{ Cooling Rate} + 543 \text{ Obtained Sr}$$

Figure 4. Tensile test result data of each sample.

Fig. 4 visualizes that obtained Sr p-value is not >0.05 , which are 0.136 for YS and 0.161 for QI. Therefore, it was obvious that on the somewhere of data pool it had to be a statistically meaningful mean difference. In order to find that, analysis of variance test (ANOVA) were performed on data subsets on Minitab 18.

On composition based data subsets, ANOVA results showed that solidification rate were impacted the results directly (Fig. 5). P-values for this data subsets were < 0.05 , therefore it can be concluded that statistical consistency was achieved. This result explains dominant behavior of cooling rate on regression analysis. On the data study of spoke number based data subset, ANOVA analysis was exposed that, the only statistically meaningful mean difference

between compositions was obtained on spoke 4, which have lowest solidification rate.

On spoke number based data subsets, ANOVA results showed that there was no statistically significant difference except than spoke 4 (Fig. 6).

Fig. 7 gives the residual plots for YS and QI. On normal distribution of residuals, it can be referred that there was no wrong regression setup mistake.

Besides, Fig. 8 shows that p-values were 0.002 for YS and 0.005 for QI. Tukey pairwise comparison on Minitab shows data grouping of ANOVA results. ANOVA sectioned the data into subsets. On YS, 4 main groups were indicated as A, AB, BC and C. On the other hand, statistically difference of mean can be satisfied on 250 ppm Sr for QI.

IV. CONCLUSIONS

Outputs of regression analysis for Sr amount and cooling rate showed that the general model for these parameters cannot satisfy tensile test outputs such as yield strength and quality index (function of tensile strength and elongation due to Equation 1).

In addition, the only statistically meaningful mean difference can be satisfied on the cooling rates which are below $0.9 \text{ }^\circ\text{C/s}$, as determined by ANOVA. It was observed that Sr amount difference cannot describe tensile test outputs. Above $0.9 \text{ }^\circ\text{C/s}$ cooling rate, composition effect is negligible, therefore solidification rate is dominant on eutectic modification which enhances tensile properties.

AUTHOR CONTRIBUTIONS

B. Dođdu performed the experiment and analyze the results. He also wrote the manuscript. O. Ertuğrul supervised this experimental study, interpret the results, and helped in manuscript preparation.

ACKNOWLEDGMENT

This work was co-supported by Tübitak 2209-B Industry / Undergraduate Thesis Support Programme and also by CMS Jant ve Makina Sanayi Company's R&D Center.

REFERENCES

- [1] G.K. Sigworth, "Fundamentals of Solidification in Aluminum Castings," *International Journal of Metalcasting*, 8(1), 7-20, 2014.
- [2] S.L. Pramod, R. Kirana, A.K. Prasada Rao, B.S. Murty, S.R. Bakshi, "Effect of Sc Addition and T6 Aging Treatment on the Microstructure Modification and Mechanical Properties of A356 Alloy," *Materials Science and Engineering A*, 674, 438-450, 2016.
- [3] S.Z. Lu, A. Hellawell, "The Mechanism of Silicon Modification in Aluminum-Silicon Alloys: Impurity Induced Twinning," *Metallurgical Transactions A*, 18, 1721-1733, 1987.
- [4] M.D. Hanna, S.Z. Lu, A. Hellawell, "Modification in the Aluminum Silicon System," *Metallurgical Transactions A*, 15, 459-469, 1984.
- [5] D. Ferdian, J. Lacaze, I. Lizarralde, A. Niklas, A.I. Fernandez-Calvo, "Study of the Effect of Cooling Rate On Eutectic Modification in A356 Aluminium Alloys," *Materials Science Forum*, 765, 130-134, 2013.

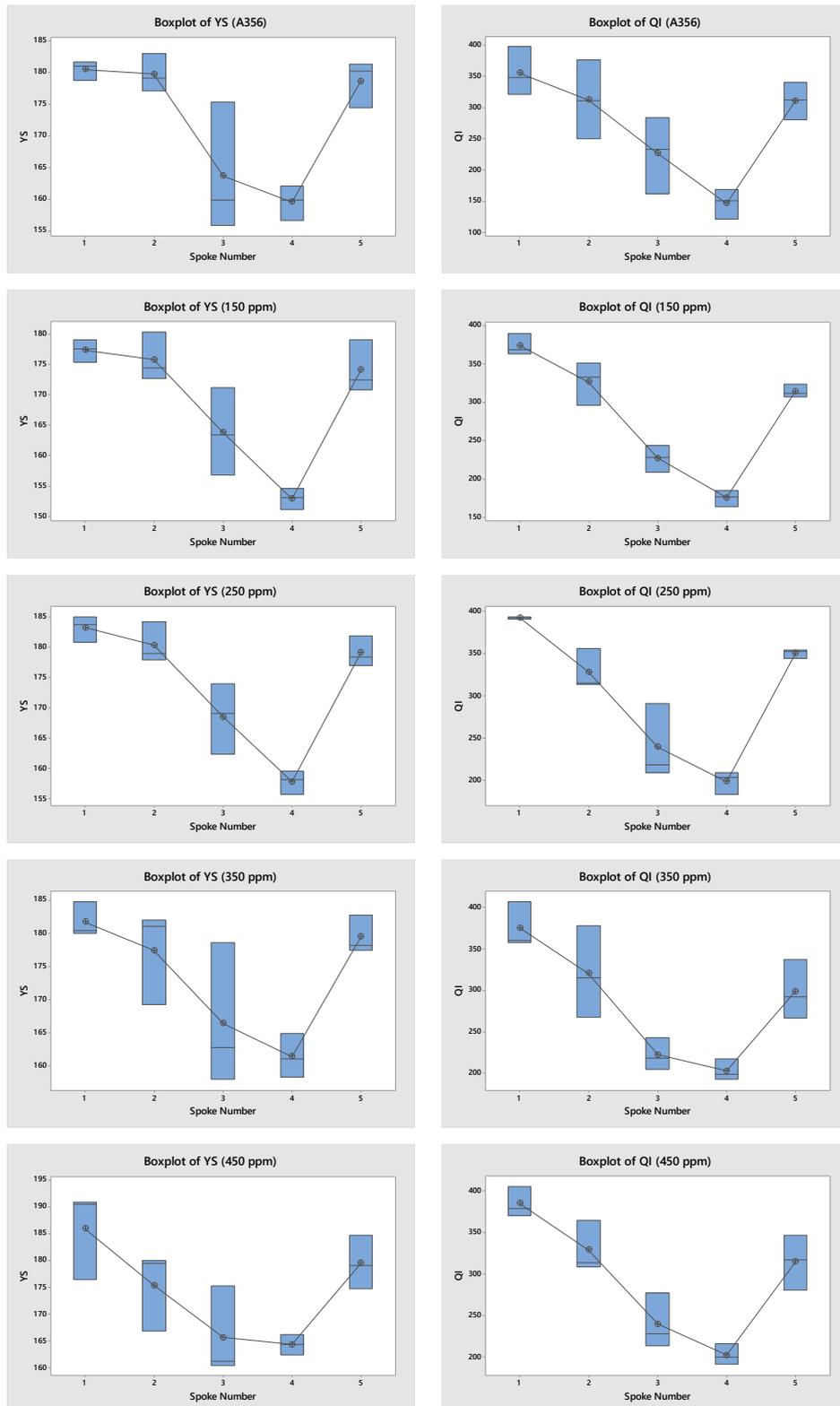


Figure 5. Boxplot of YS and QI on composition based subset.

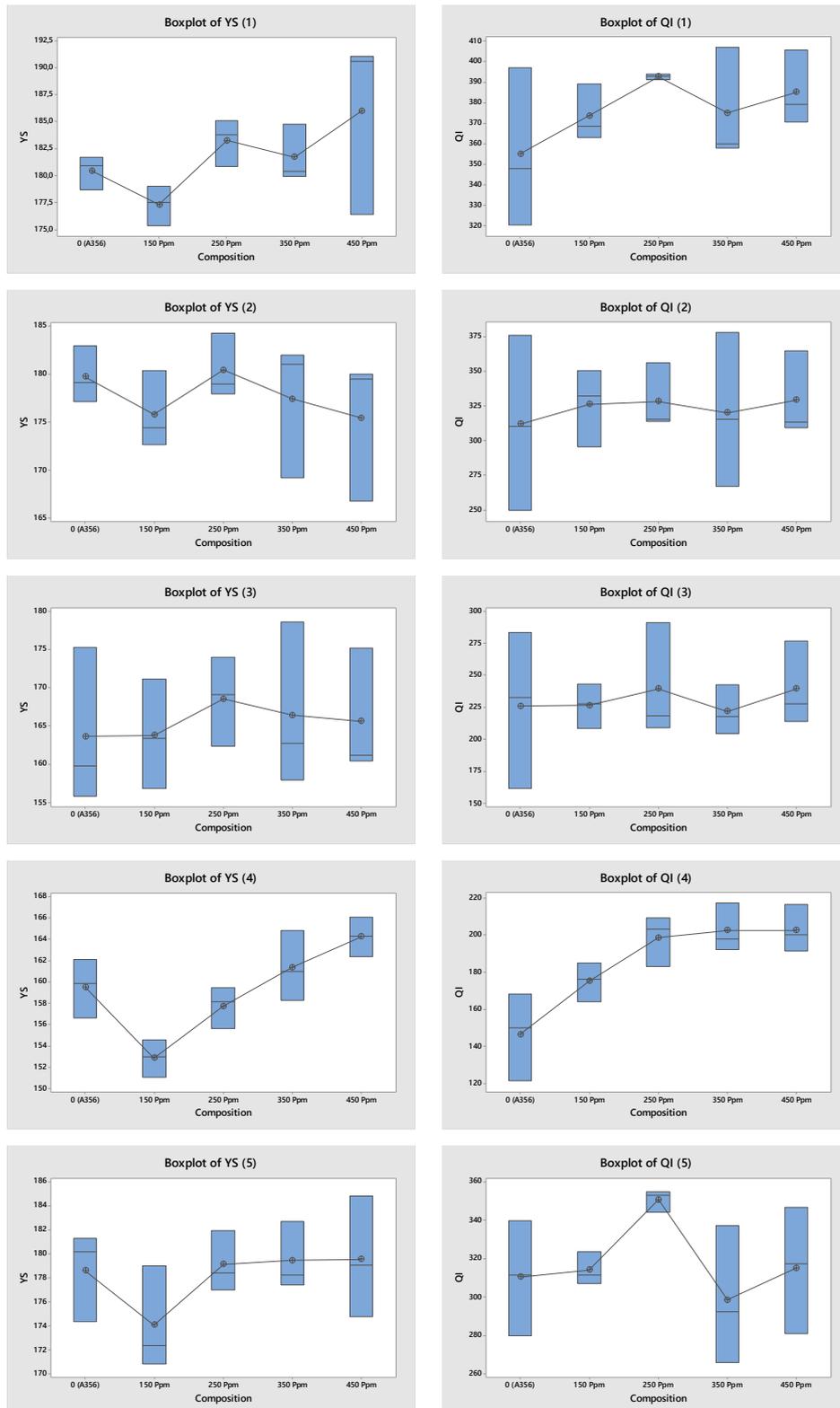


Figure 6. Boxplot of YS and QI on spoke number based subset.

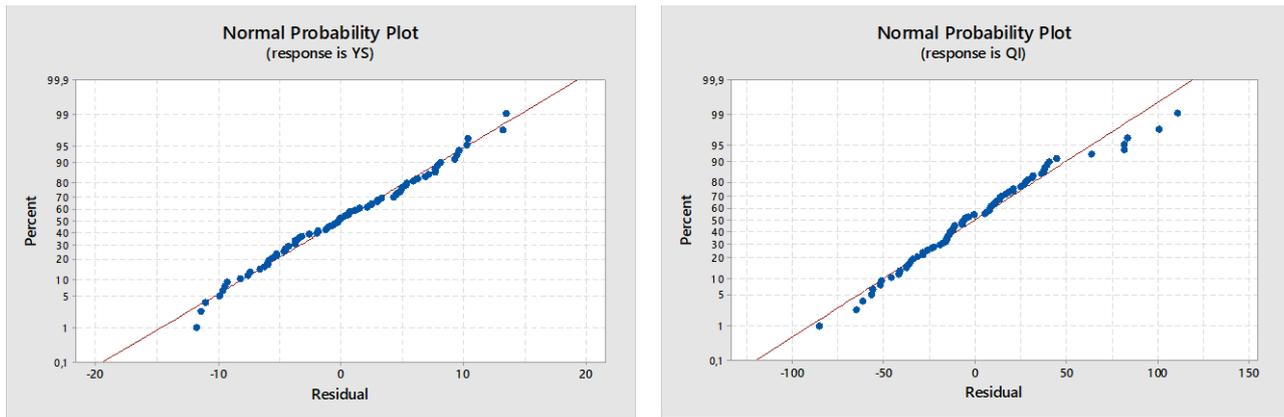


Figure 7. Probability plots of residuals for YS and QI.

One-way ANOVA: YS versus Composition

Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal
Significance level $\alpha = 0,05$

Equal variances were assumed for the analysis.

Factor Information

Factor	Levels	Values
Composition	5	0 (A356); 150 Ppm; 250 Ppm; 350 Ppm; 450 Ppm

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Composition	4	217,59	54,398	9,46	0,002
Error	10	57,51	5,751		
Total	14	275,10			

Model Summary

S	R-sq	R-sq(Adj)	R-sq(Pred)
2,39809	79,10%	70,73%	52,96%

Means

Composition	N	Mean	StDev	95% CI
0 (A356)	3	159,52	2,74	(156,44; 162,61)
150 Ppm	3	152,87	1,75	(149,78; 155,95)
250 Ppm	3	157,74	1,96	(154,65; 160,82)
350 Ppm	3	161,36	3,29	(158,27; 164,44)
450 Ppm	3	164,25	1,87	(161,17; 167,34)

Pooled StDev = 2,39809

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

Composition	N	Mean	Grouping
450 Ppm	3	164,25	A
350 Ppm	3	161,36	A B
0 (A356)	3	159,52	A B
250 Ppm	3	157,74	B C
150 Ppm	3	152,87	C

Means that do not share a letter are significantly different.

One-way ANOVA: QI versus Composition

Method

Null hypothesis All means are equal
Alternative hypothesis Not all means are equal
Significance level $\alpha = 0,05$

Equal variances were assumed for the analysis.

Factor Information

Factor	Levels	Values
Composition	5	0 (A356); 150 Ppm; 250 Ppm; 350 Ppm; 450 Ppm

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Composition	4	7142	1785,4	7,48	0,005
Error	10	2388	238,8		
Total	14	9530			

Model Summary

S	R-sq	R-sq(Adj)	R-sq(Pred)
15,4535	74,94%	64,92%	43,62%

Means

Composition	N	Mean	StDev	95% CI
0 (A356)	3	146,4	23,6	(126,5; 166,2)
150 Ppm	3	174,97	10,59	(155,10; 194,85)
250 Ppm	3	198,41	13,66	(178,53; 218,29)
350 Ppm	3	202,35	13,29	(182,47; 222,23)
450 Ppm	3	202,51	12,76	(182,63; 222,39)

Pooled StDev = 15,4535

Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

Composition	N	Mean	Grouping
450 Ppm	3	202,51	A
350 Ppm	3	202,35	A
250 Ppm	3	198,41	A
150 Ppm	3	174,97	A B
0 (A356)	3	146,4	B

Means that do not share a letter are significantly different.

Figure 8. ANOVA results of YS and QI on spoke 4.

- [6] P. Ramamurty Raju, S. Rajesh, B. Satyanarayana, K. Ramji, "Statistical Analysis of Fatigue Life Data of A356.2 Aluminum Alloy," *Structural Durability & Health Monitoring*, 7(1&2), 139-152, 2011.
- [7] S. Fintova, R. Konecna, G. Nicoletto, "Statistical Description of Largest Pore Size in Modified Al-Si Alloys," *Materials Engineering*, 16(3), 24-28, 2009.
- [8] N.D. Alexopoulos, M. Tiryakioglu, A.N. Vasilakos, S.K. Kourkoulis, "The Effect of Cu, Ag, Sm and Sr Additions on the Statistical Distributions of Si Particles and Tensile Properties in A357-T6 Alloy Castings," *Materials Science and Engineering: A*, 604, 40-45, 2014.
- [9] N.R. Green, J. Campbell, "Statistical Distributions of Fracture Strengths of Cast Al-7Si-Mg Alloy," *Materials Science and Engineering: A*, 173(1-2), 261-266, 1993.
- [10] A.M. Samuel, J. Gauthier, F.H. Samuel, "Microstructural Aspects of the Dissolution and Melting of Al₂Cu Phase in Al-Si Alloys During Solution Heat Treatment," *Metallurgical and Materials Transactions A*, 27, 1785-1798, 1996.