

Article

DOE-Based Investigation of Microstructural Factors Influencing Residual Stress in Aluminum Alloys

Nawon Kwak and Eunkyung Lee *

Interdisciplinary Major of Maritime AI Convergence, Department of Advanced Materials Convergence Engineering, National Korea Maritime and Ocean University, Busan 49112, Republic of Korea; knw010724@g.kmou.ac.kr

* Correspondence: elee@kmou.ac.kr; Tel.: +82-(051)-410-4353

Abstract

Residual stresses generated during the casting process significantly affect the reliability of the final product, making accurate prediction and analysis of these stresses crucial. In particular, to minimize the difference between simulation results and actual measurements, it is essential to develop predictive simulations that incorporate microstructural characteristics. Therefore, in this study, residual stress prediction simulations were conducted for aluminum components manufactured by high-pressure die casting (HPDC), and measurement locations were selected based on the simulation results. Subsequently, the microstructural characteristics at each location (Si and intermetallic compounds) were quantitatively analyzed, and significant factors affecting residual stress were identified through design of experiments (DOE). As a result, Si sphericity (p -value ≤ 0.05) was observed to be the most significant factor among Si area fraction, IMC area fraction, and Si sphericity, and the residual stress and Si sphericity showed a positive interaction due to the rapid cooling rate and inhomogeneous microstructure distribution. Furthermore, the study demonstrated the effectiveness of DOE in clearly distinguishing the significance of variables with strong interdependencies.

Keywords: Al-Si alloy; high pressure die casting (HPDC); residual stress; simulation; design of experiments (DOE); microstructure



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1. Introduction

The Al-Si alloys, due to their excellent castability, thermal conductivity, and high strength-to-weight ratio, are used in high-pressure die casting (HPDC) to manufacture components such as pistons, suspension and brake parts, and wheels, which require high toughness and strength [1–6]. However, during the casting process, non-uniform temperature distribution within the component leads to variations in solidification and cooling rates, resulting in the formation of residual stresses [7,8]. In particular, HPDC components, which experience rapid cooling, are more susceptible to the localized development of residual stresses [9]. These residual stresses can induce surface and internal cracks in the cast parts, leading to issues such as reduced fatigue life, distortion, and deterioration of corrosion resistance [10–12]. Moreover, residual stresses can accumulate during service, causing unexpected failures that compromise product reliability and result in significant economic losses [13,14].

Residual stress refers to the stress remaining in a material without external loads or temperature changes, and since it is an unavoidable byproduct of the manufacturing

process, accurately predicting and analyzing its distribution is important [15–17]. With advancements in simulation technologies, methods that predict the location and distribution of residual stresses based on solidification and heat treatment behaviors have recently gained attention.

However, the predictive accuracy of current simulation models is limited for aluminum alloys, as they do not account for microstructural changes that occur during the casting process [7,18]. Kianfar et al. [19] and Nikawa et al. [8] compared residual stresses measured by neutron and X-ray diffraction with simulated values, observing that the agreement between the two varied depending on the measurement location. Accordingly, microscale simulations incorporating microstructural characteristics are being developed to minimize the differences between simulation results and actual measurements [20,21].

Consequently, strategies to improve prediction accuracy have been proposed through comparisons with various simulations and the development of new numerical models [22–24]. However, systematic analyses to identify the key factors influencing residual stress are still needed. Previous studies have utilized DOE to statistically analyze the mechanical properties affected by casting parameters and heat treatment conditions of aluminum alloys, and to establish optimal process conditions [25–28]. Most studies have focused on process variables affecting mechanical properties and microstructural evolution, while DOE-based research selecting quantitative microstructural characteristics as key factors remains limited.

Therefore, in this study, residual stress measurement locations were selected based on predictive simulations, and surface residual stresses were measured using a portable X-ray residual analyzer employing the $\cos \alpha$ method. Subsequently, after grinding off the surface, DOE was employed to systematically identify the microstructural factors that have the greatest influence on the remaining residual stresses. The results of this study are expected to serve as foundational data for the future development of microscale simulation models.

2. Materials and Methods

2.1. Materials

The component used in this study was manufactured by high-pressure die casting (HPDC) and has overall dimensions of 86 cm $\times$ 41 cm $\times$ 6 cm (width $\times$ length $\times$ height), with a shape similar to that of a dog-bone. The chemical composition was analyzed using laser-induced breakdown spectroscopy (Z-903 GeoChem, SciAps, Andover, MA, USA) at the Eco-friendly Shipbuilding Core Research Support Center, National Korea Maritime and Ocean University. Each measurement was performed 10 times, and the average values for each element are presented in Table 1.

Table 1. Chemical composition of HPDC components.

Elements	Si	Mn	Mg	Fe	Cu	Ti	Ni	Sr	Al
wt%	14.44	0.84	0.34	0.27	0.17	0.10	0.01	0.01	Bal.

2.2. Residual Stress Measurement Methods

In order to predict the distribution of residual stress generated during the solidification and cooling process of the casting, a simulation was conducted using the MAGMA software (version 6.1) based on the actual geometry of the casting product. Conditions similar to the actual casting process were applied, as summarized in Table 2. In this simulation, microstructural changes during solidification and cooling were not considered. Based on this, the areas where residual stresses are expected to be concentrated in the actual casting component were derived, and the measurement area was divided into five zones.

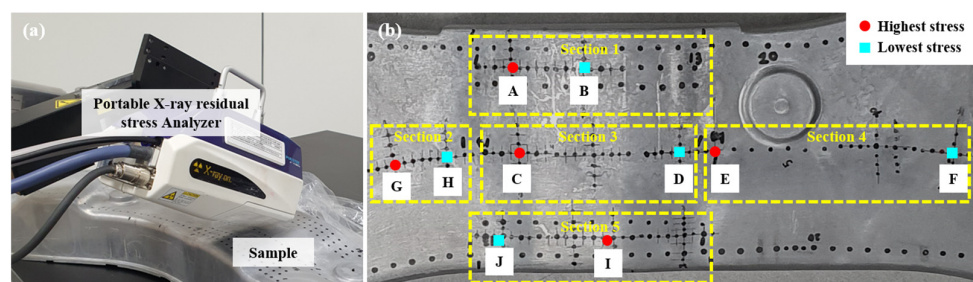
Table 2. Process conditions applied during the simulation.

Casting Parameters	Value
Cast material	Silafont-36
Die material	SKD-11
Cast temperature	700 °C
Die temperature	200 °C
Cooling condition	Water Quenching, 20 °C, 60 s

The residual stresses on the sample surface were analyzed using a Portable X-ray Residual Stress Analyzer (μ -X360s, Pulstec, Shizuoka, Japan) with a single incident beam method. Cr-K α X-rays were irradiated under conditions of 25° incidence angle, 30 kV, and 1.50 mA, with each location being measured for 30 s. To derive the principal and von Mises stresses, a second measurement was conducted after rotating the specimen by 90° following the initial measurement. The two datasets (σ_x , τ_{xy} , $\sigma_x 90^\circ$, $\tau_{xy} 90^\circ$) obtained through this process were substituted into the calculation formula (Equation (1)) provided by Pulstec Industrial Co., Ltd. (Shizuoka, Japan) [29], and the maximum and minimum principal stresses were determined based on their absolute values. Subsequently, the von Mises stress was calculated using the maximum (σ_1) and minimum principal stresses (σ_2) as shown in Equation (2) [30]. The residual stress measurement zones and locations are shown in Figure 1. The locations with the highest residual stress in each zone are indicated by a red circle, while the locations with the lowest residual stress are marked by a blue square.

$$\sigma_\theta = \sigma_x \cos^2 \theta + \sigma_x 90^\circ \sin^2 \theta + \tau_{xy} \sin 2\theta \quad (1)$$

$$\sigma_v = \sqrt{\sigma_1^2 - \sigma_1 \sigma_2 + \sigma_2^2} \quad (2)$$

**Figure 1.** (a) Portable X-ray residual stress analyzer; (b) residual stress measurement zones and locations.

2.3. Microstructure Characterization

After residual stress measurement, the locations with the highest and lowest residual stresses were selected from each of the five zones, resulting in a total of 10 residual stress analysis locations. Wire Electrical Discharge Machining (NW570, Doosan Infracore, Incheon, Republic of Korea) was used to cut square specimens measuring 2 cm by 2 cm around the locations of the highest and lowest residual stress. The cut specimens were then ground using SiC paper (400–1200 grit) and polished with 3 μ m and 1 μ m diamond suspensions, followed by final polishing using an oxide polishing suspension (OP-S). To ensure consistency, all specimens were ground under the same conditions in terms of time, applied pressure, and rotational speed, and the grinding depth was measured to be 1 mm. The residual stress measurement locations were marked before and after cutting and polishing to ensure that the microstructural analysis was performed based on the

same positions. Microstructures were observed using Optical Microscopy (Axio Imager 2, Carl Zeiss, Jena, Germany) at 1000× magnification, and image processing was conducted using ImageJ software (version 1.8.0). The obtained images were processed using Contrast Limited Adaptive Histogram Equalization (CLAHE) to uniformly adjust the brightness, and despeckle was applied to minimize noise. Subsequently, the threshold function was applied to binarize the images, with the threshold adjusted to represent the α -Al phase in white and the Si phase in black. Intermetallic compounds, which have similar contrast and brightness to the Si phase, were manually identified, as they could not be distinguished by the software. Finally, the area fraction and sphericity of the microstructural features were measured using i-Solution DT software (version 26.5).

2.4. Design of Experiments (DOE)

A two-level full factorial design was performed utilizing Minitab 22 Statistical Software to evaluate highly significant factors based on a p -value of at least 95% confidence level. In a 2-level full factorial design, there are only two levels of factors (e.g., +1, −1), resulting in 2^K runs when k factors are considered [31]. In this study, a 3-factor 2-level design was performed, the number of replicates was set to 7 to use all data, and the order of the 56 experiments was randomized. The microstructural images from the previously selected measurement locations were divided into four quadrants. The main factors were selected as Si area fraction, Intermetallic compounds (IMC) area fraction, and Si sphericity, which were obtained from the divided images. As shown in Table 3, the values were coded as +1 for values greater than the median of each factor and −1 for values less than the median. The response variable used was the residual stress remaining after the surface was ground.

Table 3. The median value of the three factors.

Factors	Si Area Fraction	IMC Area Fraction	Si Sphericity
Median	24.06	1.25	0.634

Statistical significance was evaluated using the p -value (probability value), which measures the degree of discrepancy between the observed data and the null hypothesis. A p -value smaller than the predefined significance level (α) indicates statistical significance, while a p -value greater than or equal to α means that the results are not statistically significant [32,33]. The regression equation is derived as shown in Equation (3), where y represents the dependent variable, a is the constant, and b_k and X_k denote the value (slope) of the term and factors, respectively [34].

$$y = a + b_1X_1 + b_2X_2 + \cdots + b_kX_k \quad (3)$$

3. Results and Discussion

3.1. Analysis of Residual Stress Distribution Through Simulation

A residual stress prediction simulation was conducted using MAGMA software (version 6.1) under conditions similar to the actual process. As shown in Figure 2, residual stresses were concentrated at the top and bottom areas, while high residual stresses were observed only in certain regions at the center. Based on these results, the measurement zones of the actual cast product were divided into five areas, and the surface residual stresses of each zone were measured. Subsequently, microstructural factors that were expected to influence residual stress were selected, and their correlations were evaluated using statistical methods.

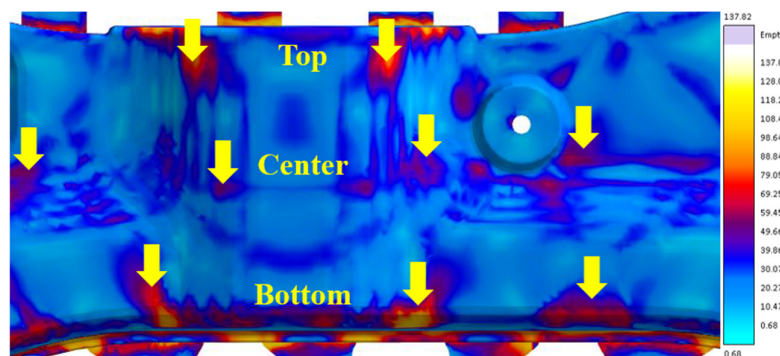


Figure 2. Predicted residual stress distribution simulation results. Yellow arrows indicate regions of residual stress concentration in the top, center, and bottom areas.

3.2. Surface Residual Stress Measurement Results

Based on the residual stress prediction simulation presented in Figure 2, the locations with the highest residual stress (A, C, E, G, I) and the lowest residual stress (B, D, F, H, J) were selected within five zones where stress concentration was expected, as shown in Figure 1. In order to minimize the influence of macroscopic structure on the residual stress and analyze the influence of microstructure alone, the residual stress after polishing was measured, and the results are shown in Figure 3. After polishing the surface, the highest residual stress was observed at location J, with a value of 59.7 MPa, while the lowest residual stress was found at location E, with a value of 11.4 MPa. Additionally, locations G and H exhibited similar levels of residual stress, at 36.5 MPa and 35.5 MPa, respectively. As shown in Figure 4, a comparison of the microstructural characteristics between the locations with the highest (J) and lowest (E) residual stress revealed that location J exhibited a higher Si area fraction, IMC area fraction, and Si sphericity than location E. These microstructural differences suggest that they may have contributed to the development of localized residual stresses. The distribution of residual stress appeared uneven depending on the measurement location, and this is presumed to be due to microstructural inhomogeneity, measurement error, and differences in surface condition, even though the same polishing conditions were applied.

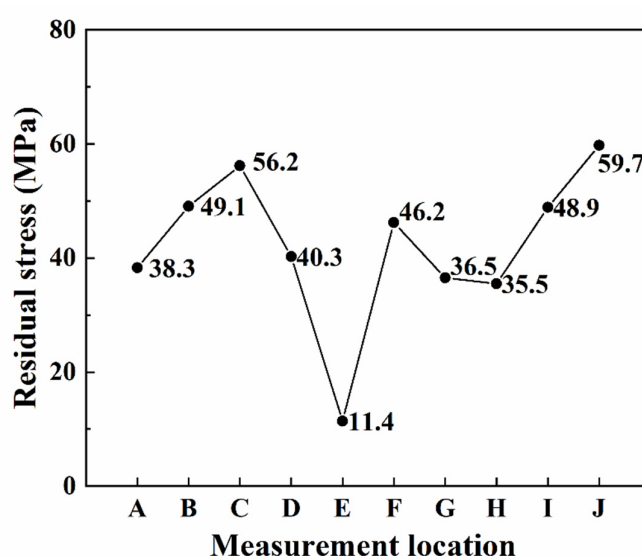


Figure 3. Residual stress measurements based on the measurement location.

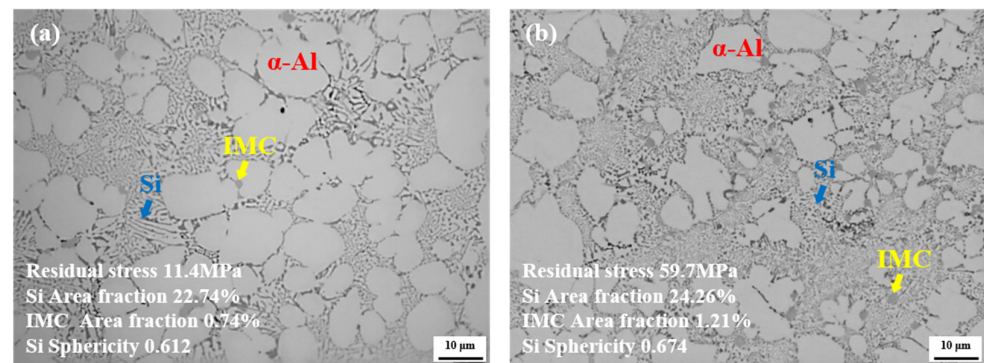


Figure 4. Optical microscopy images at residual stress measurement locations: (a) location E; (b) location J.

3.3. Results of the Design of Experiments (DOE)

To identify the main factors influencing residual stress, a 2-level full factorial design was conducted. Table 4 presents the coded results based on the median value of each factor. However, due to the uneven microstructural characteristics, bias occurred during the coding process, and certain combinations of factors (i.e., one factor being high while the other is low) did not appear in the actual data. As a result, these combinations were missing from the full factorial design. These values were not replaced with alternative values and were excluded from the statistical analysis.

Table 4. Results of the DOE, coded for use in a two-level full factorial design.

Factors					Response				
Runs	Si Area Fraction (%)	IMC Area Fraction (%)	Si Sphericity	Residual Stress (MPa)	Runs	Si Area Fraction (%)	IMC Area Fraction (%)	Si Sphericity	Residual Stress (MPa)
1	+1	+1	+1	38.28	29	+1	−1	−1	59.73
2	−1	−1	−1	11.39	30	+1	+1	+1	56.16
3	+1	+1	−1	56.16	31	+1	−1	−1	* 1
4	−1	+1	−1	40.26	32	−1	+1	+1	* 1
5	−1	−1	−1	46.22	33	−1	−1	−1	46.22
6	+1	+1	+1	38.28	34	+1	+1	−1	* 1
7	−1	−1	+1	48.88	35	−1	+1	−1	46.22
8	+1	−1	−1	38.28	36	−1	+1	−1	36.54
9	+1	−1	+1	38.28	37	1	−1	−1	* 1
10	−1	+1	−1	40.26	38	−1	−1	−1	35.47
11	−1	+1	+1	36.54	39	−1	−1	+1	* 1
12	+1	+1	−1	36.54	40	−1	+1	+1	* 1
13	+1	−1	+1	49.06	41	−1	−1	−1	35.47
14	−1	+1	+1	59.73	42	−1	−1	1	* 1
15	+1	+1	−1	35.47	43	+1	−1	−1	* 1
16	+1	−1	+1	49.06	44	−1	1	+1	* 1
17	−1	−1	+1	48.88	45	−1	1	+1	* 1
18	+1	+1	+1	49.06	46	+1	−1	+1	49.06
19	−1	−1	+1	48.88	47	+1	+1	−1	* 1

Table 4. Cont.

Factors					Response				
Runs	Si Area Fraction (%)	IMC Area Fraction (%)	Si Sphericity	Residual Stress (MPa)	Runs	Si Area Fraction (%)	IMC Area Fraction (%)	Si Sphericity	Residual Stress (MPa)
20	−1	−1	−1	46.22	48	−1	−1	−1	* 1
21	−1	+1	−1	40.26	49	1	−1	−1	* 1
22	−1	−1	+1	59.73	50	+1	−1	+1	11.39
23	−1	+1	−1	40.26	51	+1	+1	+1	36.54
24	−1	+1	+1	35.47	52	+1	+1	−1	* 1
25	−1	−1	+1	* 1	53	+1	+1	−1	* 1
26	+1	+1	+1	56.16	54	+1	−1	+1	48.88
27	−1	+1	−1	11.39	55	+1	−1	+1	59.73
28	+1	−1	−1	11.39	56	+1	+1	+1	56.16

¹ No data available for this combination.

Subsequently, backward elimination, a method of stepwise regression analysis, was applied by sequentially removing the factor with the highest p -value within the same interaction level until all remaining factors exhibited p -values less than or equal to 0.05 [35]. Factors with p -values of 0.05 or lower were considered statistically significant.

After eliminating all interaction effects among the factors, a statistical analysis considering only the main effects was performed, and the results are presented in Figure 5 and Table 5. An analysis of the primary factors—Si area fraction, intermetallic compound (IMC) area fraction, and Si sphericity—revealed that Si sphericity, which exhibited the steepest slope, had the greatest influence on residual stress, whereas the Si area fraction had the least effect. Among the three major factors, Si sphericity, with a p -value of 0.046, was identified as the most statistically significant factor.

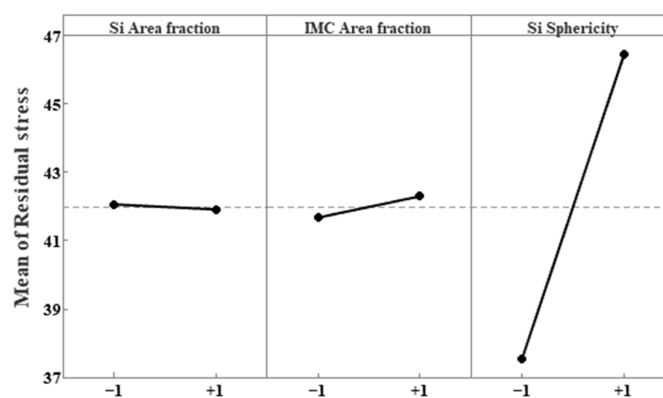


Figure 5. Main effect plots for residual stress. The dashed line indicates the reference line.

Table 5. Two-level factorial design results for a single factor.

Term	Effect	Coefficients	Standard Error Coefficients	T-Value	p -Value
Si Area fraction	−0.15	−0.07	2.15	−0.03	0.973
IMC Area fraction	0.62	0.31	2.02	0.15	0.879
Si Sphericity	8.91	4.45	2.16	2.07	0.046

The regression Equation (4) was derived through stepwise regression analysis. It was found that residual stress increases with higher Si sphericity and IMC area fraction and decreases with increasing Si area fraction.

$$\text{Residual stress} = 41.98 - 0.07 \text{ Si Area fraction} + 0.31 \text{ IMC Area fraction} + 4.45 \text{ Si Sphericity} \quad (4)$$

Additionally, although residual stress is influenced by various factors such as cooling rate and material composition, the model was simplified by sequentially removing factors with high p -values to identify the most significant factors within the experimental design of this study. The results are presented in Table 6. Si sphericity was found to be the most significant factor, with a p -value of 0.030, and a positive correlation between Si sphericity and residual stress was confirmed through the main effect analysis shown in Figure 6 and the regression Equation (5).

$$\text{Residual stress} = 41.98 + 4.41 \text{ Si Sphericity} \quad (5)$$

Table 6. Results of identifying the most significant factors using a two-level factorial design.

Term	Effect	Coefficients	Standard Error Coefficients	T-Value	p-Value
Si Sphericity	8.83	4.41	1.96	2.25	0.030

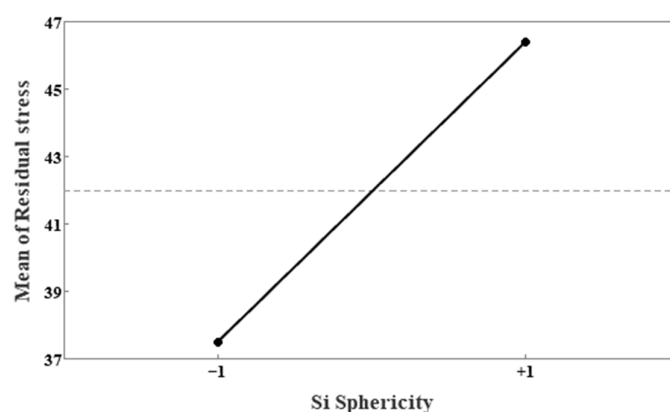


Figure 6. Main effect plot of Si sphericity on residual stress. The dashed line indicates the reference line.

3.4. Correlation Between Microstructure Factors and Residual Stresses

In this study, a high Si sphericity indicates that a rapid cooling rate was applied during the process, leading to the formation of a large number of Si particles that are close to a spherical shape. Generally, the correlation between the morphology of Si and the degree of stress concentration is such that the more spherical the shape, the lower the stress concentration, and the sharper the shape, the higher the stress concentration [36]. However, the present results show that stress is concentrated in regions with high Si sphericity, which is contrary to the general theory regarding the residual stress generated by Si sphericity within the microstructure. Unlike previous studies that analyzed changes in Si sphericity according to cooling conditions after heat treatment [37,38], this study performed water quenching immediately after casting and focused on the relationship between the shape of Si at the time of its initial formation and residual stress. Comparing location J, where residual stress is the highest, and location E, where it is the lowest, as presented in Figure 4, Si sphericity is higher at location J (with faster cooling rate), and the Si particles appear in a fine fibrous form due to Si formation. In contrast, location E (with a slower cooling rate) shows lower sphericity, and plate-like or flake Si particles were observed. This suggests that

Si became more spheroidized in regions where it had less time to form during cooling after casting. Additionally, due to the uneven distribution of microstructural factors at different measurement locations, missing data occurred during the coding process, as there was no data combination where Si area fraction was high (+1) and Si sphericity was low (−1), or Si area fraction was low (−1) and Si sphericity was high (+1). This resulted in limitations in analyzing the effect of Si sphericity alone on the residual stress within the microstructure. Additionally, both Si sphericity and fraction are influenced by the cooling rate and show similar trends, but the DOE analysis revealed that Si sphericity is a more significant factor for residual stress. Therefore, even though the two factors are interdependent, this suggests that the DOE method can statistically distinguish the factors that have a more prominent impact on residual stress. In future studies, if other microstructural variables are uniformly controlled and experiments are conducted under conditions where only Si sphericity is varied, it will be possible to clearly identify the effect of Si sphericity on residual stress.

4. Conclusions

In this study, DOE was performed based on the microstructural information of the measurement locations derived from simulations to identify the key microstructural factors affecting residual stress. The results are summarized as follows:

- (1) Based on the simulation, the residual stress measurement zones and locations were selected, with the highest residual stress of 59.7 MPa observed at location J and the lowest residual stress of 11.4 MPa observed at location E.
- (2) Through DOE, the microstructural factors affecting the residual stress after surface grinding were statistically analyzed, with Si sphericity (p -value ≤ 0.05) identified as the most significant factor.
- (3) The regions where Si particles had insufficient time to grow and thus exhibited higher sphericity showed increased residual stress due to rapid cooling, and DOE allows for the evaluation of the relative significance of variables with strong correlations.

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Conflicts of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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