Journal of Mechanical Engineering *Vol 18(3), 97-111, 2021*

Experimental Analysis on Grain Growth Kinetics of SS316L Austenitic Stainless Steel

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ABSTRACT

This paper presents an investigation of austenitic stainless steel grain growth kinetics of SS316L under different heating temperature ranges and holding times. The main variables such as apparent activation energy (Q), rate constant (K), and kinetic exponent (n) were analyzed to understand the grain growth kinetics in austenitic stainless steel. The empirical procedure was developed leading to the obtainment of variables that could define the grain growth kinetics based on different temperature ranges. The heat treatment process was isothermally held using quenching and deformations dilatometer at a temperature ranging from 900 °C to 1200 °C and holding times between 30 s to 240 s. The kinetic rates were estimated using an empirical equation. Based on the observations obtained by using optical microscopy. The result shows that the grain size can be predicted at a lower temperature than 1200 °C. However, the grains show irregular growth at a recrystallization temperature of 1200 °C which leads to a difficult estimation of grain size. It was observed that the variation in values of n and K are associated with the precipitation of the different micro-alloyed elements presented in the stainless steel SS316L. It can be also concluded that the texture plays an important role in the resulting kinetics change.

ISSN 1823-5514, eISSN 2550-164X
Received for review: 2020-12-29
Accepted for publication: 2021-04-14
Published: 2021-09-15

Keywords: *Grain growth kinetics; Kinetic exponent; Rate constant; Activation energy; Austenitic stainless steel*

Introduction

Grain size in stainless steel has been recently considered related to mechanical properties and fatigue strength by [1]–[4] while the grain size related to corrosion resistance has been studied by [5]–[7]. The final grain size after recovery, recrystallization, and phase transformations, will be the last microstructure and mechanical properties of stated metals. Knowledge on metallic alloys grain growth behaviour is important to control their physical and mechanical properties. Specifically, in welding, grain growth is an important aspect that can influence the metallurgical behaviour of certain materials. Physically, the formation of a larger grain structure will concurrently be related to the loss of good mechanical properties and corrosion resistance. Along with different changes brought by welding measures, retardation of grain growth occurs at a high temperature inside the heat-affected zone (HAZ) because of the consideration of alloying and contamination components. Changes in grain size may happen during the transformation, recrystallization process as well as the temperature influence. As stated by a previous researcher, a finer grain will increase the corrosion resistance, while a coarser grain will also increase the corrosion resistance but with an added side effect of losing the good mechanical properties [8]. Therefore, the grain size in the austenite grain structure during the manufacturing processes can be crucial, which is worth investigating.

To consider the phenomenon of grain growth more intently, the main spurred grain growth models were created in the mid-1950s [9]. In 1980, information for grain growth in the heat-affected zone of a weld can be gathered into a chart demonstrating the degree of various weld cycles at various focuses in the zone [10]. Since the significance of microstructure affects the mechanical properties, it is necessary to control grain structures in alloys and metals. Previous researchers have investigated the grain development conduct at a raised temperature and have endeavoured to show the recrystallization and grain growth of steels. Two models; the Potts model [11] and the Arrhenius model [12] were the most frequently used and further developed by other researchers.

In the last part of the 1980s, another methodology dependent on computer simulation was investigated and explored. The hard observation and processes during the experiment, for example, the volume change rate of specific grains can be regarded as simulation calculation. Various computer simulation was created with the advancement of computerized simulation software, the Monte Carlo Potts model was the most utilized and modified model [13]. An improved model along with topological contemplation of a nonlinear effective growth law results was additionally researched [13]. A recent investigation of this model of grain growth during multi-pass welding of 304 treated steel was concentrated theoretically and experimentally [14].

Different from the grain growth Monte Carlo Potts model, the Arrhenius grain growth model proposed by [12] is more suitable for low carbon stainless steel with different alloying elements. These steels are materials that present the additional alloying elements at the grain boundaries such as molybdenum, vanadium, titanium, or niobium [15]. Grain size monitoring at elevated temperature by precipitation processes is known to have commercial significance as the microstructure greatly affects the mechanical properties of the material. Many studies on the kinetic energy of the grain growth for low carbon steel and micro-alloyed steel have just been accounted for [16], [17], but few attempts have been expended to comprehend the grain growth conducted on low carbon 316 stainless steel at elevated temperature. Several studies were reporting the direct impact of grain size towards the declining corrosion resistance and mechanical properties.[5], [6], [18]–[20]. Thus, very little knowledge is known about the stated material grain growth behaviour especially at higher temperatures. This material is most familiar used in high resistance applications such as heat exchangers, jet engine parts and also suitable in marine application, in petrochemical reactor as well as gas industries.

The austenitic stainless steels contain many alloying elements which may influence grain growth. Previous studies have attempted to calculate the grain growth behaviour of SS316L material theoretically and experimentally and found that the grain growth behaviour of SS316L becomes abnormal at elevated temperatures as suggested by [21]–[23]. The development of austenite grains in micro-alloyed steels are controlled by different constituents, consider temperature and austenitization time, hot work history, chemical composition, beginning grain size and heating range at austenitization temperature.

In this research, it is aimed to investigate the grain growth of a locally sourced SS316L plate considering the initial grain size and the effect of temperature and time on the grain size development of the stated austenitic stainless steel using an empirical equation. This research will also investigate the abnormal grain growth stated by previous researchers regarding the abnormal grain growth that can occur at a temperature range of higher than 1200 °C.

Experimental Setup and Procedure

The experiment was conducted for a 4 mm SS316L austenitic stainless steel base plate. The weight percentage of the important chemical composition for the stated material is shown in Table 1. The chemical composition analysis was completed using the Bruker Q4 Tasman machine. The heat treatment was set at 900 °C, 1000 °C, 1100 °C and 1200 °C utilizing quenching and deformations dilatometers (DIL 805A/D) with holding times of 30 s, 60 s, 120 s, and 240 s followed by cooling at room temperature.

The grain growth at the Heat Affected Zone (HAZ) area was calculated using the experimental value of material activation energy and material constant which mutually describe the grain growth kinetics. The grain growth activation energy of the stated stainless-steel plate was observed. After heating at the austenitizing temperature, the sspecimens were prepared by the usual metallurgical grinding and polishing techniques combined with chemical etching. A strong chemical etching solution V2A was used to reveal the austenite grain size. Since stainless steel's chemical composition is highly resistant to corrosion, very strong acids are required to reveal its structure. Austenite grain size was calculated based on ASTM E112 standard using a single circle method measured by Leica Material Workstation.

Table 1: Chemical Composition Results (Weight percent)

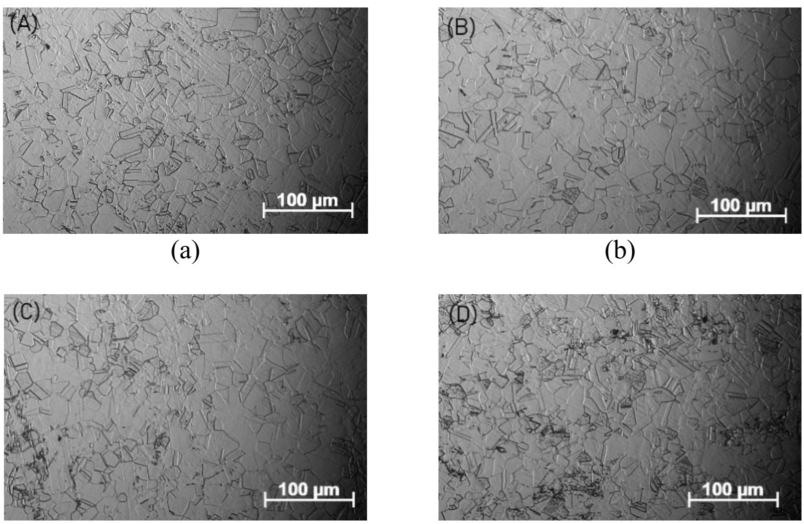
C	Si	Mn	P	S	Ni	Cr	Mo	Fe
0.017	0.34	1.47	0.02	0.069	10.19	16.76	2.148	68.44

In order to analyze the microstructure, the samples were cut parallel to the thickness. The samples were then fixed on an epoxy sample mounting, grinded using various grades of silica carbide, and polished. Using a V2A solution of 100 ccs of water, 100 ccs of hydrochloric acid, 10 ccs of nitric acid, and 2 ccs of *Sparbeize* solution for etching. The observations of microstructure were done using a metallurgical microscope Olympus BX60 with an image analysis system.

Results and Discussion

The distinctive micrographs of austenite grain boundaries under various heating temperatures and holding times are represented in Figure 1 to Figure 4. It is evident that from Figure 4(a), Figure 4(b), Figure 4(c), and Figure 4(d) the austenite grains steadily expand when the heating temperature rises from 900 °C to 1200 °C. As stated in previous studies, observable grain growth can be seen only at a temperature over 900 °C for most steels [21]. The rate of grain growth increases as the temperature rises, but there are several influences that delay the kinetics of growth. This can also be called retardation of grain growth.

The presence of alloying elements that can prevent the mobility of the grain boundaries movement is a common factor. These particles are basically very small particles of sulphides, nitrides, carbides, or silicate [24]. Austenitic stainless steel with a low carbon content that includes chromium content within it can enhance molybdenum in inhibiting recrystallization and retardation of grain growth. This is very useful commercially as it can enhance the microstructure of the stated stainless steel as a smaller grain size will provide a better mechanical property. To achieve reasonable mechanical integrity of low carbon austenitic stainless steel the heat treatment or the thermal history should be identified precisely to achieve a near-optimal grain size.

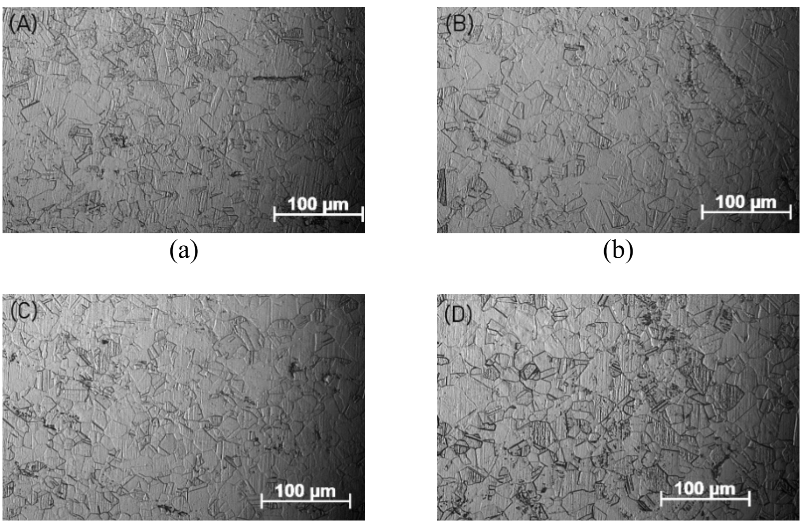


(a) (b)

(c) (d)

Figure 1: Austenite grain boundaries micrograph under various conditions of heat treatment: (a) 900 °C, 30 s; (b) 900 °C, 60 s; (c) 900 °C, 120 s; (d) 900 °C, 240 s.

Detailed precipitation studies have been investigated in austenitic stainless steel by previous researchers that indicate four distinct stages of precipitation. The four stages are its grain coarsening, initiation of grain boundary precipitation, σ phase, and finally $M_{23}C_6$ (chromium carbide) precipitation [25]. The first precipitation stage was noted at 500 °C after 61 minutes. This clearly indicates that the precipitation in austenitic stainless steel requires enough time to materialize. This was also previously investigated under the Time-Temperature-Sensitization diagram for austenitic stainless steel. The carbon content will be the major chemical composition that is related to the precipitation of the stated material at a certain temperature range. During the experimental investigation, it was observed that the second, third, and fourth stages of precipitation did not occur as the time was too rapid to allow the precipitation to happen and the temperature range varies outside the precipitation temperature range. This has allowed the investigation of the grain growth on the stated material without any obstacle or retardation. Thus, the free grain growth model proposed by previous researchers can be investigated further for the prediction that can be integrated with numerical simulation or modelling.



(a) (b) (c) (d)

Figure 2: Austenite grain boundaries micrograph under various conditions of heat treatment: (a) 1000 °C, 30 s; (b) 1000 °C, 60 s; (c) 1000 °C, 120 s; (d) 1000 °C, 240 s.

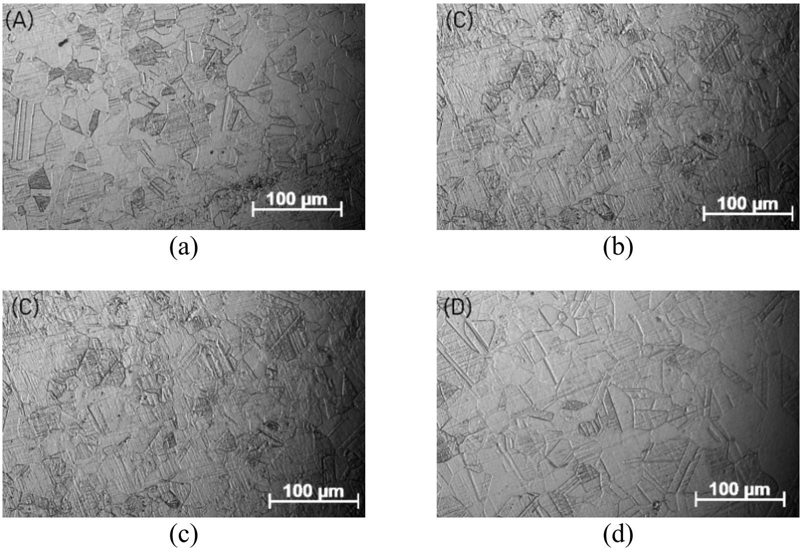
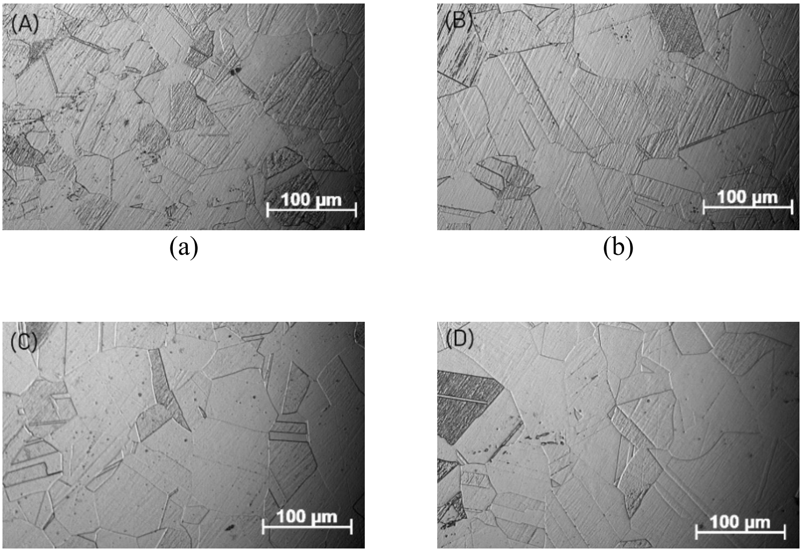


Figure 3: Austenite grain boundaries micrograph under various conditions of heat treatment: (a) 1100 °C, 30 s; (b) 1100 °C, 60 s; (c) 1100 °C, 120 s; (d) 1100 °C, 240 s.

It demonstrates that larger austenite grains would be combined with smaller ones and progressively expanded with the increasing temperature. The foremost difference can be seen in Figure 4(d), as it shows the grain boundaries movement was growing abnormally. The findings are close to those of other materials recorded previously [26], [27]. The grain size was measured and shown in Table 2.

Table 2: Average grain size (µm)

Temperature	**Holding Time**			
	30 s	60 s	120 s	240 s
900 °C	20	25	27	30
1000 °C	28	33	35	37
1100 °C	40	43	47	50
1200 °C	63	94	113	131



(a) (b) (c) (d)

Figure 4: Austenite grain boundaries micrograph under various conditions of heat treatment: (a) 1200 °C, 30 s; (b) 1200 °C, 60 s; (c) 1200 °C, 120 s; (d) 1200 °C, 240 s.

As seen in Figure 5, at both temperature and holding time, the grain size *d* of the heat-treated SS316L stainless steel plate is increased. From the normal grain growth theory proposed by previous researchers, after primary recrystallization, the normal grain growth formula form is expressed in the simplified kinetic form:

$$D = (Kt)^n \tag{1}$$

where D is the average grain size, t is the holding time, K is a constant grain growth rate and n is the grain growth exponent.

According to Arrhenius form, the constant K depends on the activation energy and the temperature. While K can be expressed with the inclusion of Q the apparent activation energy. It can be defined in the following form:

$$K = k_0 \, exp\,(-\frac{Q}{RT}) \tag{2}$$

where T is the temperature, k_0 is a constant, and R a constant with a value of 8.314 J/mol.K.

The grain size data for the stainless-steel plate SS316L was formulated using a simple kinetic model Equation (1). As presented in Figure 6, the constant grain growth exponent *n* is attained by the gradient of the straight line. The exponent of grain growth *n* shows a declining trend appropriately towards the increase of temperature, while the constant grain growth rate increases. As shown in Table 3, the predicted values of *K* and *n* were calculated. From the result, the grain growth rate decline at higher temperatures varying from 900 °C to 1200 °C. Using Equation (2) the association of value *K* and the activation energy value were obtained. The curve of the grain growth rate constant shows a linear connection with the decreasing value of temperature. Consequently, from Figure 7 the activation energy value for bulk SS316L stainless steel with a 4 mm thick base plate was estimated to be 62 kJ/mol to 110 kJ/mol. The value obtained was similar to the research done by previous researchers on the same SS316L austenitic stainless steel. This will be very interesting for welding simulation applications such as Wire-Arc Additive Manufacturing (WAAM) that has garnered a lot of interest recently with the advantage of a high deposition rate compared to the traditional machining process. This will allow the prediction of the stated material grain size that has been calibrated towards the temperature distribution history of the welding process.

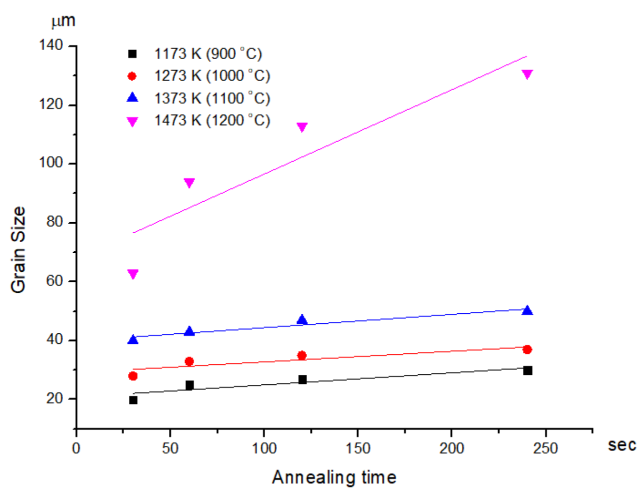


Figure 5: Isothermal grain growth kinetics at temperatures of 900 °C to 1200 °C.

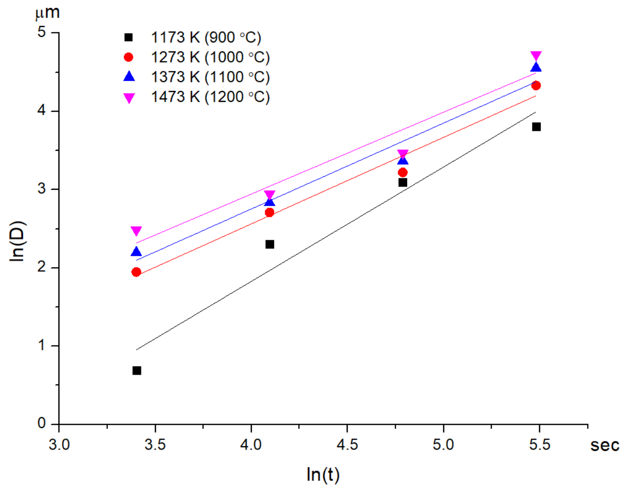


Figure 6: Grain size and holding time at selected temperatures.

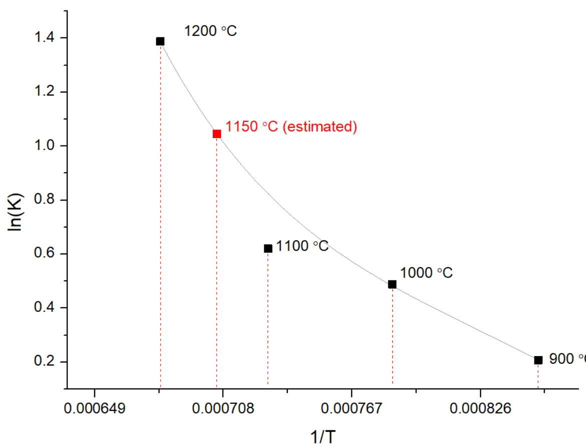


Figure 7: Growth rate constant relationship at different temperatures.

The temperature, time, chemical composition, and sample form are the main factors influencing grain growth. The activation energy for austenitic stainless steel of SS316L was previously stated at a value of 52 kJ/mol [22], comparable to the result obtained. This exhibits the chemical composition of the stated bulk material within the specimen has a significant effect on the result, as the carbon content was significantly high. Another previous study of HSLA low carbon steel has indicated a mean activation energy value of 88 kJ/mol at temperatures greater than or equal to 1200 °C, similarly, predicted by Atkinson [17], [28]. This indicates the activation energy required at a higher temperature will be lower due to the abnormal grain growth condition for this specific material, while the presence of microalloying elements by second phase particles such as carbides through precipitation is of considerable commercial importance.

Table 3: *K* and *n* values for the 4 mm SS316L base plate

Temperature	K	n	Q
900 °C	1.23	1.46	110 kJ/mol
1000 °C	1.63	1.10	96 kJ/mol
1100 °C	1.86	1.09	84 kJ/mol
1200 °C	4.01	1.04	62 kJ/mol

Previous studies also highlight that commercial FEM software with user subroutine capability [29], [30] can integrate the grain growth kinetics calculation within its numerical simulation.

Conclusion

The grain behaviour of austenite grains in SS316L stainless steel was examined by the isothermal annealing test on a quenching and deformations dilatometer machine. In view of the experimental results, the impact of heating temperature and holding time on grain size has been addressed and certain conclusions can be summarised as follows.

i. The normal grain growth formulae proposed for calculating the grain size for stainless steel either underestimates or overestimates the kinetic exponent (n), rate constant (K), and apparent activation energy (Q) value due to its inability to account for undissolved micro alloyed elements effects on austenite grain growth.
ii. Microstructure observation indicates that the grain growth mechanism of SS316L austenitic stainless steel is from grain boundary migration and abnormal grain growth occurs at elevated temperature equals to or higher than 1200 °C.
iii. The predicted value of the grain growth activation energy for SS316L stainless steel 4 mm base plate was 62 kJ/mol - 110 kJ/mol, comparable to a previous study of 52.7 kJ/mol – 88.9 kJ/mol also using the temperature range of 900 - 1200 °C [22]. The maximum error percentage is 24% at the higher temperature range due to the abnormal grain growth and different percentage of material chemical composition from the previous research.

iv. The apparent activation energy (Q_{app}) should be considered as a temperature dependant value for austenitic stainless steel as the existence of microalloying elements will affect the grain growth behaviour of the material.

As for further recommendation, the kinetic exponent (n), rate constant (K), and apparent activation energy (Q_{app}) will be considered as a temperature dependant value for further investigation on the austenitic grain growth using numerical computation. The prediction of the grain size using the same model and material with numerical computation software for the welding process will be further developed. As most of the value stated above from previous researchers was also collected from numerous resources, it was necessary to verify the local source material grain growth kinetics value as it can have different chemical compositions that can affect the grain growth. This research will continue to further investigate the same material at different temperature ranges and holding times to obtain the near-optimal condition of the grain size while exploring the mechanical and fatigue properties changes due to the grain size effect that is very important for the stainless-steel industry.

Acknowledgment

The finance of this research was supported by the German Academic Exchange Service (DAAD) with Project Code: 57525437 (Future Technology Additive Manufacturing). The authors acknowledged their gratitude to the staff members of Smart Manufacturing Research Institute (SMRI) at Universiti Teknologi MARA (UiTM), Malaysia. The authors would like to also acknowledge with gratitude for the experiment that was carried out at TU Chemnitz in Germany as well as the Professorship of Virtual Production Engineering for encouraging the research. The authors also appreciated the infrastructure provided by the School of Mechanical Engineering, College of Engineering, Universiti Teknologi MARA (UiTM).

References

[1] Y. Wang, M. Ding, Y. Zheng, S. Liu, W. Wang and Z. Zhang, "Finite-element thermal analysis and grain growth behavior of HAZ on argon tungsten-arcwelding of 443 stainless steel," *Metals*, vol. 6, no. 4, pp. 1-16, 2016.

[2] S. Zaman, M. A. Khattak and M. N. Tamin, "Effects of Welding on the Microstructural Properties of AISI 430 Ferritic Stainless Steel," *Akademia Baru*, vol. 1, no. 1, pp. 25–32, 2018.

[3] A. Turnbull and E. R. De Los Rios, "The Effect of Grain Size on Fatigue Crack Growth in an Aluminium Magnesium Alloy," *Fatigue & Fracture of Engineering Materials and Structure*, vol. 18, no. 11, pp. 1355-1366, 1995.

[4] J. Moravec, I. Novakova, J. Sobotka and H. Neumann, "Determination of Grain Growth Kinetics and Assessment of Welding Effect on Properties of S700MC Steel in the HAZ of Welded Joints," *Metals*, vol. 9, no. 6, pp. 707, 2019.

[5] R. V. Taiwade, R. Shukla, H. Vashishtha, A. V. Ingle and R. K. Dayal, "Effect of Grain Size on Degree of Sensitization of Chrome-Manganese Stainless Steel," *ISIJ International*, vol. 53, no. 12, pp. 2206–2212, 2014.

[6] S. X. Li, Y. N. He, S. R. Yu and P. Y. Zhang, "Evaluation of the effect of grain size on chromium carbide precipitation and intergranular corrosion of 316L stainless steel," *Corrosion Science*, vol. 66, pp. 211–216, 2013.

[7] C. R. Cunningham, J. Wang, V. Dhokia, A. Shrokani and S. T. Newman, "Characterisation of Austenitic 316LSi Stainless Steel Produced by Wire Arc Additive Manufacturing with Interlayer Cooling," *Proceedings of the 30th Annual International Solid Freeform Fabrication Symposium – An Additive Manufacturing Conference*, pp. 426-439, 2019.

[8] P. Bajaj, A. Hariharan, A. Kini, P. Kürnsteiner, D. Raabe and E. A. Jägle, "Steels in additive manufacturing: A review of their microstructure and properties," *Materials Science & Engineering A*, vol. 772, no. 138633, 2020.

[9] J. E. Burke and D. Turnbull, "Recrystallization and grain growth," *Progress in Metal Physics.*, vol. 3, no. C, pp 220-292, 1952.

[10] M. F. Ashby and K. E. E. Easterling, "A first report on diagrams for grain growth in welds," *Acta Metallurgica*, vol. 30, pp. 1969–1978, 1982.

[11] A. Ayad, F. Wagner, N. Rouag and A. D. Rollett, "Accelerated Potts model for grain growth - Application to an IF steel," *Computational Material Science*, vol. 68, pp. 189-197, 2013.

[12] I. Andersenj and O. Grong, "Analytical modelling of grain growth in metals and alloys in the presence of growing and dissolving precipitates. I- Normal grain growth," *Acta Metallurgica,* vol. 43, no. 7, pp. 2673–2688, 1995.

[13] D. Zöllner and P. Streitenberger, "Normal grain growth: Monte Carlo Potts model simulation and mean-field theory," *Micro-Macro-interaction in Structured media and Particle Systems.*, pp. 3–18, 2008.

[14] X. Chen, X. Chen, H. Xu and B. Madigan, "Monte Carlo simulation and experimental measurements of grain growth in the heat affected zone of 304 stainless steel during multipass welding," *International Journal of Advance Manufacturing Technology.*, vol. 80, no. 5, pp. 1197-1211,

2015.
[15] T. T. Gladman and D. Dulieu, "Grain-Size Control in Steels," *Metal Science*, vol. 8, no. 1, pp. 167-174, 1974.
[16] J. Moon, J. Lee and C. Lee, "Prediction for the austenite grain size in the presence of growing particles in the weld HAZ of Ti-microalloyed steel," *Material Science and Engineering A*, vol. 479, no.1, pp. 40-46, 2007.
[17] S. Illescas, J. Fernández and J. M. Guilemany, "Kinetic analysis of the austenitic grain growth in HSLA steel with a low carbon content," *Materials Letters*, vol. 62, no. 20, pp. 3478–3480, 2008.
[18] X. H. Chen, J. Lu, L. Lu and K. Lu, "Tensile properties of a nanocrystalline 316L austenitic stainless steel," *Scripta Materialla*, vol.52, no. 10, pp. 1039-1044, 2005.
[19] X. Feaugas and H. Haddou, "Grain-size effects on tensile behavior of nickel and AlSl 316L stainless steel," *Metallurgical and Materials Transactions: Physical Metallurgy and Materials Science,* vol. 34 A, no. 10, pp. 2329–2340, 2003.
[20] G. Dini, A. Najafizadeh, R. Ueji and S. M. Monir-Vaghefi, "Tensile deformation behavior of high manganese austenitic steel: The role of grain size," *Materials and Design*, vol. 31, no. 7, pp. 3395–3402, 2010.
[21] D. Priadi, R. A. M. Napitupulu and E. S. Siradj, "Austenite grain growth calculation of 0.028% Nb steel," *Journal of Mining and Metallurgy Section B: Metallurgy*, vol. 47, no. 2, pp. 199–209, 2011.
[22] B. P. Kashyap and K. Tangri, "Grain growth behaviour of type 316L stainless steel," *Materials Science and Engineering A*, vol. 149, no. 2, pp. 13-16, 1992.
[23] M. Faiz Mat, Y. H.P. Manurung, N. Muhammad, S. N. S. Ahmad, M. Graf and M. S. Sulaiman, "Variable Analysis for Grain Size Prediction of Austenitic Stainless Steel SS316l Using Heat Treatment," *Journal of Applied Science*, vol. 20, no. 3, pp. 91–96, 2020.
[24] R. Abbaschian, L. Abbaschian and R. Reed-Hill, "Physical Metallurgy Principles", in Annealing, 4th ed. Cengage Learning, USA, 2009, pp. 216-260.
[25] D. N. Wasnik, G. K. Dey, V. Kain and I. Samajdar, "Precipitation stages in a 316L austenitic stainless steel," *Scripta Materialla*, vol. 49, no. 2, pp. 135–141, 2003.
[26] M. Shirdel, H. Mirzadeh and M. H. Parsa, "Abnormal Grain Growth in AISI 304L Stainless Steel," *Materials Characterization*, vol. 97, pp 11-17, 2014.
[27] R. C. Chen, C. Hong, J. J. Li, Z. Z. Zheng and P. C. Li, "Austenite grain growth and grain size distribution in isothermal heat-treatment of 300M steel," *Procedia Engineering*, vol. 207, pp. 663–668, 2017.
[28] H. V. Atkinson, "Overview no. 65. Theories of normal grain growth in pure single phase systems," *Acta Metallurgica*, vol. 36, no. 3, pp. 469-

491, 1988.

[29] O. Yahya, Y. H. P. Manurung, M. S. Sulaiman and P. Keval, “Virtual Manufacturing for Prediction of Martensite Formation and Hardness Value induced by Laser Welding Process using Subroutine Algorithm in MSC Marc / Mentat,” *Journal of Mechanical Engineering*, vol. 15, no. 2, pp. 107–125, 2018.

[30] Y. H. P. Manurung, M. S. Sulaiman, S. K. Abas, G. Tham and E. Haruman, “Investigation on welding distortion of combined butt and T-joints with 9-mm thickness using FEM and experiment,” *International Jornal of Advance Manufacturing Technology*, vol. 77, no. 5, pp. 775-782, 2015.