Parametric Optimisation of Heat Treated Recycling Aluminium (AA6061) by Response Surface Methodology

A. Ahmad[1, a)], M. A. Lajis[1, b)], N.K. Yusuf[1, c)], S. Shamsudin[1, d)], and Z.W. Zhong[2, e)]

[1] *Sustainable Manufacturing and Recycling Technology, Advanced Manufacturing and Materials Centre (SMART-AMMC), Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, Batu Pahat, Johor, Malaysia.*

[2] *School of Mechanical and Aerospace Engineering, Nanyang Technological University, Singapore, Singapore.*

Corresponding author: a)amri@uthm.edu.my
b)azlan357@gmail.com
c)nurkamilahyusuf@gmail.com
d)shazarel@uthm.edu.my
e)mzwzhong@ntu.edu.sg

Abstract. Alternating typical primary aluminium production with recycling route should benefit various parties, including the environment since the need of high cost and massive energy consumption will be ruled out. At present, hot extrusion is preferred as the effective solid-state recycling process compared to the typical method of melting the swarf at high temperature. However, the ideal properties of extruded product can only be achieved through a controlled process used to alter the microstructure to impart properties which benefit the working life of a component, which also known as heat treatment process. To that extent, this work ought to investigate the effect of extrusion temperature and ageing time on the hardness of the recycled aluminium chips. By employing Analysis of Variance (ANOVA) for full factorial design with centre point, a total of 11 runs were carried out randomly. Three dissimilar extrusion temperatures were used to obtain gear-shape billet. Extruded billets were cut and ground before entering the treatment phase at three different ageing times. Ageing time was found as the influential factor to affect the material hardness, rather than the extrusion temperature. Sufficient ageing time allows the impurity atoms to interfere the dislocation phenomena and yield great hardness. Yet, the extrusion temperatures still act to assist the bonding activities via interparticle diffusion transport matter.

INTRODUCTION

Mining ore from the earth in an uncontrolled way is a very irresponsible action. Cutting open pits in natural landscape to strip mining for bauxite not only contributes to deforestation, but also results in a very serious pollution to the human being. Such typical mining operation to produce so-called “virgin” alloys could jeopardise the environment, as it had already terminally ill, due to the reckless human activities. Therefore, an alternative to help to reduce the earth austerity should be emphasized in order to preserve healthier environment for future generation. Recycling of aluminium had been discussed for many years back from the 1900s. Several recycling techniques had been practised which in turn met the objective of serving back to the consumer either for the same purpose or a slightly different in the function. While deliberating on the recycling technique, it finally comes to a conclusion that the method should incorporate with low consumption of energy and effective cost in its implementation when compared to the pure alloy mining from the bauxite. Till now, there are still debating among researchers on the most effective technique of aluminium recycling.

Several reports deduced that there are still metal lost at every stage of the conventional recycling process. The losses had been contributed by the metal oxidation during melting, burning, metal mixed with the slag from the surface of the melt, and others are scraps resulted from casting and further processing of the aluminium ingots [1]–[4]. As an alternative to conventional technique which required the alloy to be completely liquefied, thus, the emerging of solid-state recycling technique has been embraced to eliminate the liquid state. Instead of melting the alloy by means of high temperature, solid-state recycling technique practices more environmental friendly approach [5]. The waste was directly recycled through appropriate extrude metal forming operation at

3rd Electronic and Green Materials International Conference 2017 (EGM 2017)
AIP Conf. Proc. 1885, 020046-1–020046-7; doi: 10.1063/1.5002240
Published by AIP Publishing. 978-0-7354-1565-2/$30.00

temperatures somewhat above the recrystallization temperature. Such recycling practice is relatively simple, consumes small amounts of energy and does not have a harmful effect on the environment [6]–[9].

Secondary alloys production usually yields the material performance slightly below than the virgin alloy strength. To that extent, the anticipated mechanical properties of extruded product can be obtained only by means of a final heat treatment. Optimising both solution and ageing heat treatment given to such alloys could reveal better secondary material performance. Through heat treatment process, it is able to provide material strengthening by precipitation hardening mechanisms [10], [11].

As key process in this study, the heat treatment was implemented over the aluminium chip-based extrudate. The effect of different ageing time at different extrusion temperatures was studied to acquire a proper relationship between the two factors. Since there are no studies reporting the influence of such factors on the material hardness, the correlation between the factors is hoped to be clarified statistically through the analysis of variance (ANOVA) by employing response surface methodology (RSM) analysis.

EXPERIMENTAL PROCEDURE

Material preparation

The investigated alloy 6061 – AlMgSi bulk was provided by commercial vendors. The bulk was inspected through Energy Dispersive Spectroscopy (EDS) analysis by using EMAX Horiba that was attached to a Hitachi SU1510 SEM. The element was confirmed to comply and follow the Standard Specification for Aluminium and Aluminium-Alloy Sheet and Plate [12]. The chemical composition of the alloy matrix used in the present work consists of Si (0.17 wt.%), Mg (5.84 wt.%), Al (93.98 wt.%) and the balance fraction remains for the other elements.

To mimic the waste produced from the machining process, the waste chip was prepared by milling the as-cast alloy through certain cutting speed, feed and depth of cut [13], [14]. The milling process was done using Sodick-MC430L high-speed machining. As soon they left the machining process, the chips were immediately cleaned using acetone solution. Soaking in such solution may remove the impurities from the machining process (i.e. grease). The degreasing process was done to ensure that it did not jeopardise the diffusion bonding during the solid-state recycling of the chip [15]. Assisting the process with vibration from the ultrasonic bath may improve the cleaning process. The drying process took place afterwards at 60ºC in the drying furnace.

Hot extrusion process

The chips were placed in a cylindrical 30mm diameter container with a height approximately 90mm. Pre-compaction of the cleaned chips was done by a cold press machine for 30 tonne at the room temperature. The green-compacted billet was extruded according to the selected temperature and preheating period. Table 1 depicts the designated value for the extrusion process.

TABLE 1. Hot extrusion parameters

Parameter	Unit	Value/type
Extrusion die shape	-	Spur Gear
Extrusion ratio	-	4.86
Billet diameter	*mm*	30
Extrusion speed	*mm/s*	4.4
Extrusion temperature	*°C*	450, 500, 550
Heating period	*min*	120

The green-compacted billet was heated for 450, 500, 550 °C before being transferred into the extrusion container. The extrusion process was carried out through a hydraulic press, having a maximum capacity of 300 tonne and the plunger moved forward to extrude the billet at 4.4 mm/s. The leaving billet out of the die will take the shape of the spur gear with the dimensions as given in Table 2.

TABLE 2. Dimensions of spur gear die

Parameter	Unit	Value/type
Gear type	-	Spur
Module	-	1.0
Number of teeth	-	14
Pitch diameter	*mm*	24
Pressure angle	*o*	20

At the instant the specimen left the die, it was cut and ground to the length of 10mm. Then, the specimen was heated to 530°C for 120 minutes before rapidly quenched into cold water to ensure fast cooling. Distinct ageing times were implemented as a consideration of two important issues related to heat treatment which is the selection of temperature and the duration of ageing treatment. Both are dependent on its previous chemical composition and heat treatment history [16], [17]. The flow of the heat treatment applied is shown schematically in Figure 1.

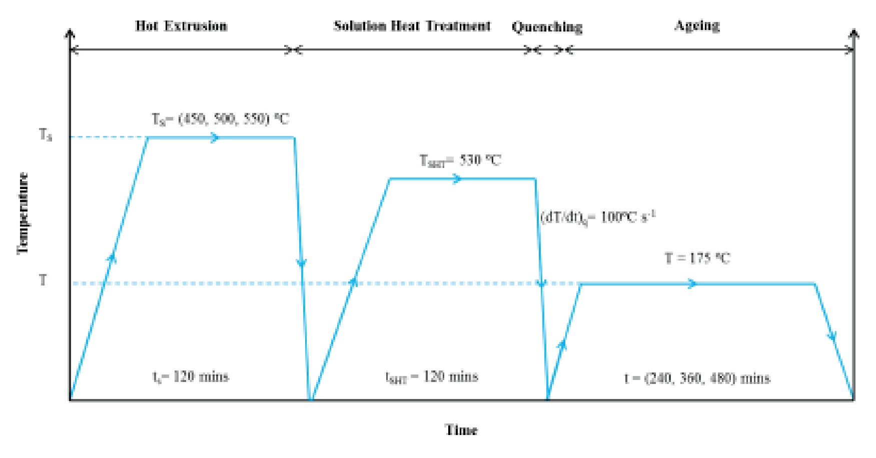

FIGURE 1. Heat treatment temperature diagram

The samples were prepared for microhardness (MH) testing by grinding consecutively on 60, 240, 400, 600 and 1200 SiC papers. The microhardness test was performed using Vickers Hardness tester (Shimadzu HMV-2TE) by forcing a square-based pyramidal diamond indenter, having face angles of 136° at 2.942 N (HV 0.3). The duration of the applied load was 10 seconds, involving 10 randomly distributed indentations on each run. The sequence of the hot extrusion process together with the heat treatment implementation is illustrated in Figure 2.

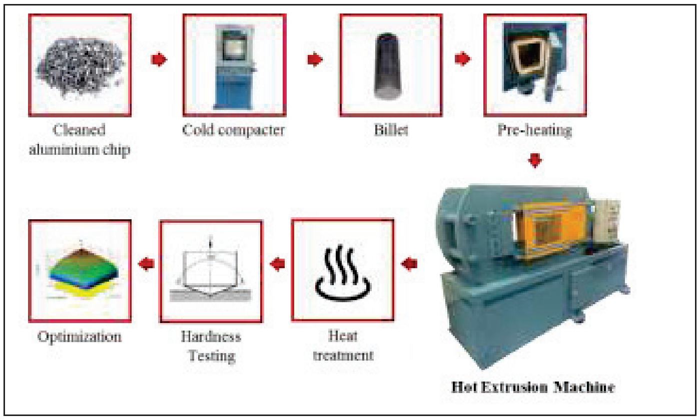

FIGURE 2. Hot extrusion process flow

Experimental design

The effects of extrusion temperature and ageing time were analysed thoroughly with responses surface methodology (RSM) by adopting central composite design (CCD) principle. The star points were located at the face of the cube portion of the design, corresponding to an α-value of 1, commonly referred to, as a face-centred, and the centre points, as implied by the name, were points with all levels set to coded 0. Single experiment was carried out at each factorial run and axial points which resulted in 8 runs. Whilst at the centre point, 3 repetitions were carried out that finally resulted in a total of 11 runs throughout the experiment. Three centre points in the design were used to test the curvature effect of the model and for development of quadratic equation if it existed. Table 3 presents the factors and levels used in this study.

TABLE 3. Factors and levels for response surface study

Factor	Parameter	Notation	Unit	Levels		
				(-1)	(0)	(+1)
A	Extrusion Temperature	T	ºC	450	500	550
B	Ageing time	t	minutes	240	360	480

The response studied through this experiment is the microhardness of the samples. Analysis of variance (ANOVA) was utilised to analyse the relationship between each parameters and also to identify the most dominant factors over response of interest. The appropriate model for predicting the optimal condition is by using the quadratic model form as shown in Equation 1 [18].

$$\mathrm{y} = \beta_0 + \sum_{j=1}^{k} \beta_j x_\mathrm{j} + \sum_{j=1}^{k} \beta_{jj} x^2{}_\mathrm{j} + \sum_{i} \sum_{<j=1}^{k} \beta_{ij} x_\mathrm{i} x_j + \varepsilon \qquad (1)$$

Where y is the response; x_i and x_j are the factors; β_0 is a constant coefficient; β_j, β_{jj} and β_{ij} are the interaction coefficients of the linear, quadratic and second-order terms, respectively; k is the number of studied factors and ϵ is the error. The fitted polynomial equation is expressed as surface and contour plots in order to visualise the relationships between the response and experimental levels of each factor and to deduce the optimum conditions [19], [20]. After optimisation, the adequacy of the model equation for predicting the optimum response values was validated with experimental results. The details of the order of experimental run are given in Table 4.

RESULT AND DISCUSSION

The results of the heat treatment on the extruded samples are tabulated in Table 4 and arranged according to standard run while all experiments were carried out randomly by following the experimental run. The results were used for further analysis by the Design Expert 8.0 software. The examined Fit Summary of the response revealed a linear model is statistically significant and therefore it was further used in correlation analysis.

TABLE 4. Sequence of the experimental runs

Std. Run	Input variable				Output
	Factors (Coded)		Factors (Actual)		Microhardness (HV)
	T	t	Extrusion Temp. (ºC)	Ageing time (minutes)	
1	-1	-1	450	240	99.68
2	+1	-1	550	240	105.66
3	-1	+1	450	480	81.89
4	+1	+1	550	480	90.64
5	-1	0	450	360	89.87
6	+1	0	550	360	102.71
7	0	-1	500	240	102.62
8	0	+1	500	480	86.48
9	0	0	500	360	96.86
10	0	0	500	360	94.84
11	0	0	500	360	95.17

The ANOVA table for response surface of a quadratic model for UTS is shown in Table 5. The f-value of 104.52 with p-value < 0.05 suggested that the model is significant. There is only a 0.01% chance that a "*Model f-Value*" is large could occur due to noise. If the p-value is less than 0.05, it showed that the corresponding model terms were significant and desirable, where their influence has considerable effect on the microhardness.

According to that, the main effect of extrusion temperature (T) and ageing time (t) were the significant model terms. Since that the p-value for Lack-of-Fit is 0.3107 and larger than 0.05, it implies that the final model is concluded to be adequate due to the insignificant in Lack-of-Fit test.

TABLE 5. ANOVA table (partial sum of square) for microhardness

Source	Sum of Squares	Degree of freedom	Mean Square	*f*-Value	*p*-value	
Model	526.03	2	263.02	104.52	< 0.0001	significant
T-Extrusion Temp.	126.68	1	126.68	50.34	0.0001	
t-Ageing time	399.35	1	399.35	158.69	< 0.0001	
Residual	20.13	8	2.52			
Lack of Fit	17.78	6	2.96	2.52	0.3107	not significant
Pure Error	2.35	2	1.17			
Cor Total	546.17	10				
Std. Dev.	1.5863					
R^2	0.9631					
Adjusted R^2	0.9539					
Predicted R^2	0.9211					
PRESS	43.0876					

Figure 3 shows the perturbation plot for microhardness. The factor of ageing time has greater influence over microhardness than the factor of extrusion temperature. Figure 4 shows the 3D surface plot with respect to the main effects of extrusion temperature and ageing time.

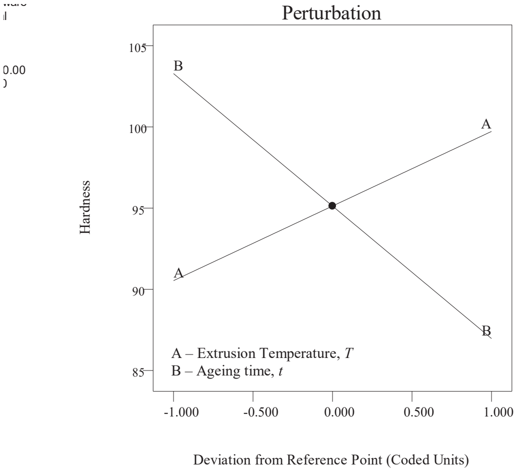

FIGURE 3. Perturbation plot for MH

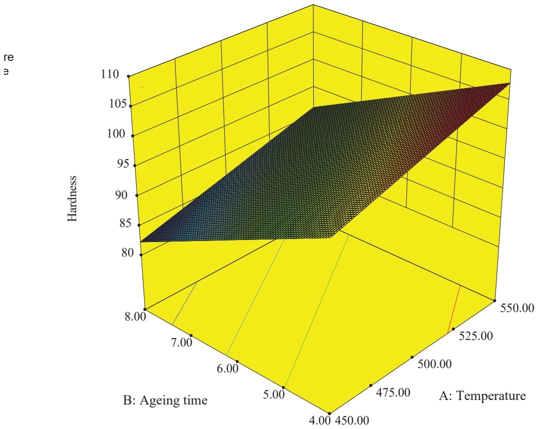

FIGURE 4. 3D contour plot for MH

RSM provides the predictive model in terms of coded and uncoded (actual) factors. Coding makes direct comparisons between coefficients possible, while the actual terms change depending on the unit of measure. The final linear equation for microhardness is given as follows;

$$MH = 95.13 + 4.59\,T - 8.16\,t \tag{2}$$

The model shows an adequate representation of the microhardness response. By comparing Equations 2 with Equation 1, one would notice that $\beta_2 > \beta_1$ for the response. It can be concluded that the ageing time had the greatest effect on the responses due to its highest main effect value.

The ageing time dominantly influences the findings. The best properties in hot extrusion parts of heat-treatable aluminium alloys are obtained by including heat treatment in the forming cycle that provides strengthening by precipitation hardening mechanisms [11]. Precipitation of a second phase (solute rich particles) from the matrix is normally used to increase strength, hardness and other mechanical properties [10], [16]. These second phases are metastable and therefore transformed into equilibrium phase with sufficient ageing time. The highest temperature setting of 550°C resulted in the highest hardness as recorded. The individual bonding quality of chips including the grain growth strengthening and the diffusion mechanism may attribute to the material strength. Utilizing higher temperature in the extrusion process would result in finer equiaxed grains, which led to the great material performance [15], [21]–[23]. The high temperature causes the activation energy increased, allowing more atoms to fill up the pores and voids and enhanced interparticle diffusions, contributing to stronger bonding between individual chips [24]. Moreover, the chips strength is reduced when the heat is applied, which in turn intensify the ductility mode of the material. To that extent, more oxide layers will be broken up that could result in massive exposure of virgin to virgin aluminium. Therefore, in line with the improved intimate contact of chip surfaces, the bonding will be enhanced due to the increases of inter-atomic force and chips coalescence [15].

The maximum hardness exhibited was 105.66 HV at 240 minutes of aging time and extrusion temperature 550°C considered as the peak-aged. An increase in hardness could be explained by a diffusion assisted mechanism, and also by the hindrance of dislocation due to impurity of atoms, i.e. foreign particle of the second phase, since the material after quenching from 530°C (solution heat treatment) will have an excessive vacancy concentration [25]. It is also found that as the ageing time and temperature increase, the density of Guinier–Preston zones will also tend to increase [26]. Hence, the degree of irregularity in the lattices will cause an increase in the mechanical properties of the aluminium alloy. However, by proceeding to 360-480 minutes of ageing time, the value of microhardness decreased linearly. This could be due to the coalescence of the precipitates into larger particles which will cause fewer obstacles to the movement of dislocation, and also due to annealing out of the defects. This also may be due to the formation of different precipitates and over ageing of alloy as reported by many others [25], [27]–[30].

CONCLUSION

The effects of extrusion temperature and ageing time on the microhardness have been investigated using RSM.

a. The ANOVA results revealed that, ageing time was the most influential parameter, followed by extrusion temperature that was found to be less significantly affecting the response.
b. The maximum hardness recorded for 240 minutes of ageing time considered as the peak-aged. Such peak point was achieved through the diffusion activities of the matrix. Excessive vacancy present within the specimen as the material left the quenching process allows the impurity atoms to interfere the atom dislocation which yield great material hardness.
c. Higher extrusion temperature assists in improving the chip bonding, which is caused by the interparticle diffusion transport matter. High strain during the process with high billet temperature intensifies the diffusivity between the aluminium chips.
d. Over ageing is encountered when the time schedule is more than 240 minutes. This might be because the coalescence of the precipitates into larger particles caused fewer obstacles to the movement of dislocation.

ACKNOWLEDGMENTS

The authors would like to express their profound gratitude and deepest appreciation to the Ministry of Higher Education (MOHE), Malaysia, for funding this project through the Malaysian Technical University Network-Centre of Excellence (MTUN-CoE) and Fundamental Research Grant Scheme (FRGS) through grant numbers of 1496, 1463 and 1426. The authors would also like to express their special thanks to Sustainable Manufacturing and Recycling Technology, Advanced Manufacturing and Materials Centre (SMART-AMMC), Universiti Tun Hussein Onn Malaysia (UTHM) for their support.

REFERENCES

1. J. Gronostajski and A. Matuszak, J. Mater. Process. Technol. 92, 35 (1999).
2. J. Gronostajski, H. Marciniak, A. Matuszak, and M. Samuel, J. Mater. Process. Technol. 119, 251 (2001).
3. J. Gronostajski, W. Chmura, and Z. Gronostajski, J. Mater. Process. Technol. 125, 483 (2002).
4. J. Cui and H. Roven, Trans. Nonferrous Met. Soc. China 20, 2057 (2010).
5. J.R. Duflou, A.E. Tekkaya, M. Haase, T. Welo, K. Vanmeensel, K. Kellens, W. Dewulf, and D. Paraskevas, CIRP Ann. - Manuf. Technol. 64, 37 (2015).
6. A.E. Tekkaya, M. Schikorra, D. Becker, D. Biermann, N. Hammer, and K. Pantke, J. Mater. Process. Technol. 209, 3343 (2009).
7. K. Suzuki, X.S. Huang, A. Watazu, I. Shigematsu, and N. Saito, Mater. Sci. Forum 544, 443 (2007).
8. S. Wu, Z. Ji, and T. Zhang, J. Mater. Process. Technol. 209, 5319 (2009).
9. J.. B. Fogagnolo, E.. M. Ruiz-Navas, M.. Simón, and M.. Martinez, J. Mater. Process. Technol. 143, 792 (2003).

10. A. Forcellese and F. Gabrielli, Int. J. Mach. Tools Manuf. 40, 1285 (2000).
11. D.A. Tanner and J.S. Robinson, Mater. Des. 29, 1489 (2008).
12. ASTM B209-14, ASTM Int. 1 (2007).
13. N.K. Yusuf, M.A. Lajis, M.I. Daud, and M.Z. Noh, Appl. Mech. Mater. 315, 728 (2013).
14. A. Ahmad, M.A. Lajis, N.K. Yusuf, and A. Wagiman, ARPN J. Eng. Appl. Sci. 11, 2258 (2016).
15. S. Shamsudin, Z.W. Zhong, S.N.A. Rahim, and M.A. Lajis, Int. J. Adv. Manuf. Technol. 1 (2016).
16. C. Meng, Effect of Preheating Condition on Strength of AA6060 Aluminium Alloy for Extrusion, 2010.
17. J. Pezda, Metalurgija 53, 63 (2014).
18. M.H. Muhamad, S.R. Sheikh Abdullah, A.B. Mohamad, R. Abdul Rahman, and A.A. Hasan Kadhum, J. Environ. Manage. 121, 179 (2013).
19. R. Triveni, T.R. Shamala, and N.K. Rastogi, Process Biochem. 36, 787 (2001).
20. N. Salim, R. Hashim, O. Sulaiman, M. Ibrahim, M. Sato, and S. Hiziroglu, Compos. Part B Eng. 43, 988 (2012).
21. M. Haase and a. E. Tekkaya, J. Mater. Process. Technol. 217, 356 (2015).
22. Z. Sherafat, M.H. Paydar, and R. Ebrahimi, J. Alloys Compd. 487, 395 (2009).
23. Y. Chino, M. Kobata, K. Shimojima, H. Hosokawa, Y. Yamada, H. Iwasaki, and M. Mabuchi, Mater. Trans. 45, 361 (2004).
24. J. Gronostajski, J. Kaczmar, H. Marciniak, and A. Matuszak, J. Mater. Process. Technol. 64, 149 (1997).
25. H. Demir and S. Gündüz, Mater. Des. 30, 1480 (2009).
26. R.A. Siddiqui, H.A. Abdullah, and K.R. Al-Belushi, J. Mater. Process. Technol. 102, 234 (2000).
27. F. Ozturk, A. Sisman, S. Toros, S. Kilic, and R.C. Picu, Mater. Des. 31, 972 (2010).
28. B. Mirzakhani and M. Mansourinejad, Procedia Eng. 10, 136 (2011).
29. I.M. Masoud, T. Abu Mansour, and J.. Al-Jarrah, 8, 5106 (2012).
30. D.D. Risanti, M. Yin, P.E.J.R.D. del Castillo, and S. van der Zwaag, Mater. Sci. Eng. A 523, 99 (2009).