Microhardness and Microstructure of Hot Extrusion Parameters in Direct Recycling of Aluminium Chip (AA 6061) by ANOVA Method

Mohammed H Rady¹², Mohd Sukri Mustapa³, S Shamsudin⁴, M A Lajis⁵ and A Wagiman⁶

Abstract. Microhardness and microstructure become a main concern in direct recycling of metal chips. Products by solid-state recycling of aluminum chips in hot extrusion process are controlled by many factors such as die geometry, extrusion ratio, temperature related parameters and etc. in which each of them could influence the extrudate’s quality. This study investigates the effects of preheating temperature and preheating time on the response variable (microhardness and microstructure). The various settings of preheating temperature were taken as 450 °C, 500 °C, and 550 °C. On the other hand, three values of preheating time were chosen (1, 2, 3) hours. The influences of the process parameters (preheating temperature and time) were analyzed using Design of Experiments (DOE) approach whereby full factorial design with center point analysis was adopted. The total runs were 11 and they comprise of two factors of full factorial design with 3 center points. The results found that the preheating temperature is more important to be controlled rather than the preheating time in analysis of microhardness and decreasing of temperature led to the high microhardness. The profile extruded at 450 °C and 1 hr had gained the optimum microhardness and it can be concluded that setting temperature at 550 °C for 3 hour resulted in the highest responses for average of grain sizes in analysis of microstructure.

1. Introduction

Aluminum production has been extensively used for many applications accordingly recycling of aluminum prompts a tremendous number of cost and environmental benefits. In examination with various materials, aluminum production has one of the greatest energy differences between primary and secondary production at 186 MJ per 1 kg and 10–20 MJ per 1 kg respectively [1]. From this reason various manufactures have now centers of growing and expanding the utilization of secondary materials [2]. Aluminum chips produced by machining are the most champion among a wide range of scrap to reuse by remelting, as the oxidation of the material is increased in view of the high surface to volume extent of the chips [3]. A direct recycling of aluminum alloy machining chips into finished or semi-finished product is an alternative method to overcome the issue of losing material by using remelting of aluminum chips and to additionally build up the energy balance of the aluminum production [4]-[6]. Aluminum as chips can be directly changed into semi-finished or finished items through mechanical operations, for example, hot extrusion, hot forging, rolling, severe plastic deformation forms, friction extrusion, conform process and etc [1], [7]. Products by solid-state recycling of aluminum chips in hot extrusion process are controlled by many factors such as die geometry, extrusion ratio, temperature related parameters and etc. in which each of them could influence the extrudates’ quality [8], [9]. This
study investigates the effects of preheating temperature and preheating time on the response variables (microhardness and microstructure).

2. Working Procedures

2.1. Preparing the chips

The chips were preparing by comminuting the block of aluminum through high-speed milling. Selection of the milling kind was because they have a thinner shape which leads to get better deformability [8]-[10]. The chip particle’s size was examined by a Toolmaker measuring microscope outfitted with a digital Nikon MM-60 camera and are shown in Fig. 1. The obtained chips by face milling processing were curled up and discontinuous. The selected material for this study was aluminum AA6061-T6 heat-treated, which has wide applications in an industry of automotive. The mechanical properties of the aluminum alloy selected in this research are summarized in Table 1.

2.2 Cleaning and drying the chips

The chips were degreased with acetone inside an ultrasonic bath for 10 minutes for removing any impurities and dirt. The method of chips cleaning chips followed ASTM G131-96, the standard practice applicable to clean materials by ultrasonic methods. Finally, the process of drying used was with conventional drying oven for 1 hour to dry up the solution of acetone or any moisture left on the chips made earlier.

2.3 Cold press process

The cleaned machining chips were then placed in a cylindrical container and compacted at room temperature by a cold press around 80 mm. The sequence of the chips preprocessing is shown in Fig. 2.

2.4 Hot extrusion process

The billets were extruded through the assigned conditions as shown in Table 2. The ranges of preheating temperature and time selected were between 450 and 550 °C and 1 and 3 hr, respectively. The maximum preheating temperature was limited to 550 °C because the billet heating above 550 °C results in hot cracks in the surface of the extrudates, while the ram speed setting was bounded to below or equal to 1mm/s to prevent an in homogeneous flow of material due to the effect stick-slip resulting in chatter marks on the surface of profile at higher speeds [11]. The heat was created utilizing a ceramic heater installed around the container. A graphite-based lubricant was utilized over the inward surface of the container and the die at every cycle of extrusion so as to restrict the increase in extrusion load as an influence of friction.
Figure 1. Characteristics of milling type of chips and chip-based billet appearance after compaction

Table 1. Mechanical properties of AA6061-T6

| Mechanical properties | Value (all values measured under T = 25 °C) |
|-----------------------|-------------------------------------------|
|                       | Max. | Min. |
| Density (g/cm3)       | 2.70 | 2.66 |
| Tensile strength (MPa)| 319  | 315  |
| Yield strength (MPa)  | 292  | 291  |
| Elongation (%)        | 13   | 12   |

Figure 2. Preprocessing of chips before consolidation
Table 2. Parameter setting of hot extrusion process

| Parameter       | Value/type |
|-----------------|------------|
| Extrusion die   | Round      |
| Extrusion ratio R | 5.4       |
| Billet ø (mm)   | 30         |
| Extrusion speed (mm/s) | 1     |
| Container temp (°C) | 300      |
| Die temp. (°C)  | 300        |
| Preheating temp. (°C) | 450,500,550 |
| Preheating time (hr) | 1,2,3   |

2.5. Experimental design

Experimental runs based on the $2^3$ full factorial design were achieved to investigate the influences of two process parameters as previously mentioned. Three center points were included in the design to check the curvature effect of the model and the interactions between parameters were also considered. The design scheme is listed in Table 3. Only single run was executed in each corner and total 11 runs were included. The details of the experimental run are given in Table 4 for microhardness. Responses in this study were microhardness and microstructure of the fabricated samples. Analysis of variance (ANOVA) was applied to rank the main effects and to analyze the interactions between the input parameters. The DOE results can suggest an insight of further experimental direction for process optimization.

Table 3. Design scheme of process parameters and their levels

| Parameter symbol | Parameter          | Levels          |
|------------------|--------------------|-----------------|
|                  |                    | Low (-1)        |
|                  |                    | Center (0)      |
|                  |                    | High (+1)       |
| A                | Preheating temp    | 450             |
|                  |                    | 500             |
|                  |                    | 550             |
| B                | Preheating time    | 1               |
|                  |                    | 2               |
|                  |                    | 3               |

3. Result and discussions

3.1 Result of microhardness properties and discussions

Total eleven runs were carried out according to the full factorial design with three center points. The overall results of the experiments are presented in Table 4. The data shows that the minimum preheating temperature gives the high resulting microhardness.

3.1.1. Microhardness test

Microhardness testing was done at load of 0.9807 N and holding time of 10 seconds at room temperature. In this way, the hardness test was achieved by using the micro Vickers hardness machine. It is extremely useful for testing on a wide type of materials as long as samples are carefully prepared. A square base pyramid shaped diamond is used for testing in the Vickers scale. Three times of the test were investigated for each specimen. At that point, the average was taken of these values. The result of microhardness was that the extrude specimen at a lower preheating temperature is observed to be harder than the extrude specimen at a high preheating temperature, showing that the microhardness is sensitive to preheating...
temperature of the extrusion because the grain size in entire regions is large at higher temperatures while extruded specimens at lower preheating temperature are gotten to initiate that amounts of normal stress and shear strain at the area neighbouring to the die wall are high to form a hard skin on the extrudates. On the hard skin layer, the plastic flow of the chips is limited to a generally small zone underneath the indenter to realize about higher hardness.

Table 4. Results of microhardness and density

| Sample Designation | A (°C) | B (hr) | Microhardness |
|--------------------|--------|--------|--------------|
| S1                 | 1      | 450    | 50.9254      |
| S2                 | 2      | 550    | 44.9448      |
| S3                 | 3      | 450    | 49.9987      |
| S4                 | 4      | 550    | 44.2872      |
| S5                 | 5      | 450    | 55.6886      |
| S6                 | 6      | 550    | 44.3944      |
| S7                 | 7      | 450    | 50.9192      |
| S8                 | 8      | 550    | 44.3572      |
| S9                 | 9      | 500    | 44.8555      |
| S10                | 10     | 500    | 42.2784      |
| S11                | 11     | 500    | 51.4043      |

*Note: A = Preheating Temperature, B = Preheating Time

3.1.2. Analysis of ANOVA results
The ANOVA results show that the significant term contributing to microhardness in direct hot extrusion process of aluminium AA6061 is preheating temperature, it informed by p < 0.05 as shown in Table 5 and Fig. 3 (Pareto Chart). The remaining factors, preheating time and interaction of preheating temperature and preheating time are not significant.

Table 5. Results of ANOVA for microhardness

| Source DF | Adj SS | Adj MS | F-Value | P-Value |
|-----------|--------|--------|---------|---------|
| Model     | 4      | 126.185| 31.546  | 3.37    | 0.090   |
| Linear    | 2      | 114.243| 57.122  | 6.10    | 0.036   |
| Preheating Temp | 1 | 109.138| 109.138| 11.65   | 0.014   |
| Preheating Time | 1 | 5.105  | 5.105  | 0.55    | 0.488   |
| 2-Way Interactions | 1 | 3.127  | 3.127  | 0.33    | 0.584   |
| Curvature | 1      | 8.815  | 8.815  | 0.94    | 0.369   |

Figure 3. Pareto chart of microhardness
In DOE analysis, the main effect plot and interaction plot of microhardness are as shown in Fig. 4 and Fig. 5 respectively. The main effect plot is clearly shows that preheating temperature is significant parameter in which the response is influenced while preheating time is unimportant parameter toward microhardness. As can be seen in interaction plot that it indicates the same trend. It shows that the final model selected is appropriate for the observed data. On the other hand, the curvature effect is insignificant on the response and also confirmed by the ANOVA results in Table 5, where p > 0.05 for the curvature term. Therefore, linear model is enough to fit all the data.

![Main effect plot for microhardness](image)

**Figure. 4** Main effect plot for microhardness

![Interaction plot for microhardness](image)

**Figure. 5** Interaction plot for microhardness

3.1.3. Response Optimizer

The response optimizer method is used to show which factors have effect on extrudates product separately and their impact on the response variables (microhardness). This method gave the best value for these responses. Fig. 6 represents the analysis of optimizer response from (DOE) for microhardness.
4. Analysis of microstructure

The chip-extruded samples were observed by optical microscope (OM). For OM, samples were ground with SiC grits paper 240, 600, and 1200 for 180 seconds each in wet condition, polished with colloidal silica and etched utilizing Barker’s reagent with a voltage of U = 12 V DC for 120 seconds. Grain sizes were calculated using a mean linear intercept technique according to ASTM E112-13 standard.

Fig. 7 indicates the typical optical micrographs of the chip-extruded alloy prepared at various preheating temperatures and time. We can see that fine grains appeared in the microstructure of the samples preheated at 450 °C, which was because of the consequence of dynamic recrystallization during hot extrusion process but chip boundaries and sizes of grain are small, then no cracks and voids can be showed in these samples. At this temperature, the average of grain size at 1 hour and 3 hours is 18.59 and 24.77 µm respectively. With increasing preheating temperature to 500 °C, the unrecognized and nonuniform shapes were showed in case of the new microstructure. Smooth boundary lines, no voids have been observed and the specimens contained a very dense microstructure, This finding can be related with Zhao’s [12] work. At 550 °C, the shape of grains became more equiaxed and recrystallized and showing that grain coarsening had occurred at this processing temperature, similar to those commented by Shamsudin and Hasse [1,13]. The average grain size at 500 °C and 550 °C preheating temperatures were bigger than the grain size compared to the specimens preheated at 450 °C. One of the averages of grain size at 500 °C was 33.96 µm while its value at 550 °C at 1 hour and 3 hours was 31.29 and 35.28 µm respectively as shown in table 6.
Figure 7. Optical micrograph of the samples extruded from the chip-based billets at different preheating temperature and time a- 450 °C, 1 hour; b- 450 °C, 3 hours; c- 500 °C, 2 hours; d- 550 °C, 1 hour; e- 550 °C, 3 hours.
Table 6. Grain size measurement

| Sample | Preheating Condition | G no. | Average Diameter(µm) |
|--------|----------------------|-------|----------------------|
| S1     | 450 °C, 1 h          | 8.60  | 18.30                |
| S2     | 550 °C, 1 h          | 7.54  | 26.36                |
| S3     | 450 °C, 3 h          | 8.79  | 17.16                |
| S4     | 550 °C, 3 h          | 7.13  | 30.47                |
| S5     | 450 °C, 1 h          | 8.55  | 18.59                |
| S6     | 550 °C, 1 h          | 7.05  | 31.29                |
| S7     | 450 °C, 3 h          | 7.73  | 24.77                |
| S8     | 550 °C, 3 h          | 6.71  | 35.28                |
| S9     | 500 °C, 2 h          | 7.39  | 27.82                |
| S10    | 500 °C, 2 h          | 7.15  | 30.26                |
| S11    | 500 °C, 2 h          | 6.82  | 33.96                |

5. Conclusion
The study investigates intensively the effects of preheating temperature and preheating time on the resulting microhardness and microstructure of the extruded profiles in hot extrusion process of direct recycling of aluminum AA6061 wastes. The ANOVA results conclude that the factor of preheating temperature is very significant to microhardness performances but the preheating time is less influential factor towards it than the preheating temperature, then microhardness is sensitive to the extrusion preheating temperature and it can be concluded that setting temperature at 550 °C for 3 hour resulted in the highest responses for average of grain sizes in analysis of microstructure.

Acknowledgment
The authors would like to express the deepest appreciation to the Centre for Graduate Studies, Universiti Tun Hussein Onn Malaysia (UTHM). Additional support was also provided by Sustainable Manufacturing and Recycling Technology, Advanced Manufacturing and Materials Center (SMART-AMMC), Universiti Tun Hussein Onn Malaysia (UTHM).

References
[1] S Shamsudin, Z. W. Zhong, S. N Ab Rahim & M. A. Lajis (2016) The influence of temperature and preheating time in extrudate quality of solid-state recycled aluminum. Int J Adv Manuf Technol DOI 10.1007/s00170-016-9521-4.
[2] G. Gaustad, E. Olivetti, and R. Kirchain, “Economic and environmental evaluation of various aluminum scrap upgrading options using chance constrained optimization modeling,” in Global symposium on recycling, waste treatment, and clean technology (REWAS), 2008.
[3] Ahmed Sahib Mahdi, Mohd Sukri Mustapa, Mohd Amri lajis and Mohd warikh abd Rashid (2016) Compression Strength and Microhardness of Recycling Milled Aluminium (AA6061) for Various Binder Vol. 3, Issue 2, pp: (98-104), Month: October 2015 - March 2016.
[4] J. Gronostajski, H. Marciniak, and a. Matuszak, “New methods of aluminium and aluminiumalloy chips recycling,” J. Mater. Process. Technol., vol. 106, pp. 34–39, 2000.
[5] Kadir, M.I.A., Mustapa, M.S., Ibrahim, M.R., Samsi, M.A., Mahdi, A.S.(2017) Microstructures and characteristics of solid state recycling aluminium chips AA6061/Al-SiC composites fabricated by cold compaction method.
[6] Samsi, M.A., Mustapa, M.S., Badarulzaman, N.A., , Kadir, M.I.A., Mahdi, A.S. 2017 The effect of quenching on physical characteristics of recycled AA6061 aluminum chips.
[7] Mahdi, A.S., Mustapa, M.S., Lajis, M.A., Rashid, M.W.A. 2016, Effect of compaction pressure on mechanical properties of aluminium particle sizes AA6061al alloy through powder metallurgical process.

[8] Chiba R, Yoshimura M (2015) Solid-state recycling of aluminium alloy swarf into c-channel by hot extrusion. JManuf Process 17(0): 1–8. doi:10.1016/j.jmapro.2014.10.002.

[9] S Shamsudin, M. A. Lajis and Z. W. Zhong, Solid-state recycling of light metals: A review, 2016.

[10] S.N. Ab Rahim, M.A. Lajis, S. Ariffin. A Review on Recycling Aluminum Chips by Hot Extrusion Process, 2015.

[11] Sami. Al-Alimi, M. A. Lagis, S. Shamsudin. A Review on Solid-State Recycling of Light Metal Reinforced Inclusions by Equal Channel Angular Pressing, 2017.

[12] Kondoh K, Luangvaranunt T, Aizawa T (2002) Solid-state recycle processing for magnesium alloy waste via direct hot forging. Mater Trans 43(3):322–325.

[13] ZHAO Zu-de1, CHEN Qiang1, YANG Lin2, SHU Da-yu1, ZHAO Zhi-xiang1(2011) Microstructure and mechanical properties of Mg-Zn-Y-Zr alloy prepared by solid state recycling.

[14] Haase M, Ben Khalifa N, Tekkaya AE, Misiolek WZ (2012) Improving mechanical properties of chip-based aluminum extrudates by integrated extrusion and equal channel angular pressing (iECAP. Mater Sci Eng A 539(0):194–204. doi:10.1016/j.msea.2012.01.081. 