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Influence of Process Variables on The Density of Aluminium Chip-Base Feedstock Prepared for Non-Melted Hot Extrusion Recycling

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ABSTRACT

Non-melted recycling of aluminum chips through hot extrusion offers a sustainable manufacturing alternative. However, achieving void-free extrudates is crucial, as air trapped in chip-based feedstocks may cause microvoids. In this work, heat treatment was performed on the chips before they were compacted into the feedstock. The effects of annealing temperature, time, and compaction pressure on density were investigated. The experiment was conducted using a 2^3 factorial design, and an ANOVA was used to identify the main parameters. Visual inspections and testing were conducted on the compacted feedstocks. The results indicate that parameters such as compaction pressure, annealing temperature, and annealing time are statistically significant. The main parameter is the compaction pressure, followed by the annealing temperature, the interaction between the annealing temperature and the annealing time, and finally the annealing time. At high annealing temperatures, annealing time does not significantly affect density, but at low temperatures, annealing time plays a crucial role. Non-annealed chip feedstocks exhibit lower relative density compared to annealed ones. A higher feedstock density leads to a reduction in surface voids on the final product. This work highlights the importance of heat treatment and compaction pressure in achieving quality extrudates, thus advancing the non-melted recycling technique for sustainable manufacturing.

1. Introduction

Aluminium chips generated from the machining process typically have a higher value than scrap from post-consumer products. This is because the chips are less contaminated and have a high purity level. The scrap can be recycled without melting, resulting in lower energy costs and a more environmentally friendly process. A variety of severe plastic deformation techniques can be used to recycle aluminium chip without melting. The technique includes direct hot extrusion [1,2] indirect

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hot extrusion [3], forging [4,5] and rolling [6] have been proven viable in transform aluminium scrap into finish or semi-finish product

Recycling aluminium chips using hot extrusion can be accomplished either using powder metallurgy route or chip-based route [7]. The process may include chip separation, comminution, cleaning, compaction and extrusion. Post process such as heat treatment and mechanical means [8,9] may also be applied to improve the properties of the extrudates. The heat treatment on extrudates were performed to obtain homogeneous grain microstructure. While in cyclic extrusion and rolling, the grain size is reducing in providing more homogeneous structure. The process also eliminates microvoid defect, making the distance between the surface chip close contact and permit solid diffusion among the surface.

Recycling of aluminium by direct hot extrusion is always associated with microvoid defects [10], which are caused by air that trapped in porous compacted chip-based feedstock [11]. Feedstock with high porosity would result in extrudate with significant microvoids. Besides that, chips will easily disintegrate and difficult in transferring during the process. The porosity of the feedstock could be measured from its density. The density of the feedstock is influenced by the compaction process variable such as the technique, compaction pressure and time [12-16].

Previous research introduced various technique in producing high density compacted chip-based feedstock. Tokarski *et al.,* [17] found that single layer compaction at compaction pressure of 240 MPa could produce feedstock density of 77 %. While Hasse and Tekkaya [18] compacted the chips at 303 MPa result in feedstock density of 82 %. Misiolek *et al.,* [19] introduced multi-layer compaction in order to increase the density of the feedstock. Although the multi layer compaction could result in higher density, however the feedstock is easily crack at the inter layer. This is due to the interface between the former and the subsequence layer has poor consolidation. The reasons are that the compaction has developed strain hardening at the former layer, thus make the subsequence chips compaction hard to diffuse into the former layer. Besides that, the chips also have a distinctive level of strain hardening resulted from the machining process itself [20]. Therefore, compaction at high pressure is required to obtain high shear strain that enough to break the aluminium oxide on the chip surface. However, compaction performed at higher pressure would reduce the tool life and increase the operation cost

The chips can be reduced in strength by annealing it before undergo compaction process. This technique was introduced by Samuel [21] and Kore *et al.,* [9]. They concluded that there is no doubt that the annealing treatment is capable of softening aluminum chips and increasing the density of the feedstock. However, studies that explain the influence of compaction and the annealing process variables are seldom reported. Annealing treatment on the chip before the compaction process is an alternative to reduce the strength of the chip. The technique has been introduced by Samuel [21] and also applied by Kore where they concluded there is no doubt that the annealing treatment can soften the aluminium chips and improve the density of the feedstock. However, research work explains the effect of the annealing parameters on the density is seldom reported. Therefore, this research was conducted to determine the influence of compaction and annealing parameters on density. The study is necessary as annealing involves a heating process which reflects the operating cost.

2. Methodology

Figure 1 illustrates the process of making the chip-based feedstock. The chip was prepared by mill machining on a square billet of AA6061-T651 as to simulate the industrial aluminium scrap. The chemical compositions of the material are tabulated in Table 1. The mill machining was carried out by using MAZAK 3-axis CNC with a cutting condition of side cut end mill, 10 mm cutting tool diameter, 345 mm/second cutting speed, 1 mm feed per tooth and 1 mm depth of cut. An oil-water emulsion was used as a coolant during the cutting. The chips were cleaned in an ultrasonic acetone bath for 30 minutes to remove the oil-water emulsion that fouled on the chip surface. The clean chips are then dried in the furnace oven at 100 ^{0}C for 1 hour to vaporize the acetone and water. The annealing process was carried out by heating the chip in the furnace at 300– 500 ^{0}C to remove the strain hardening developed during machining and homogenize the chip microstructure. The chips were left to cool down naturally in 10 minutes. Within this time, the chip has reached the ambient temperature of 26 ^{0}C to 28 ^{0}C.

Fig. 1. Chip-based feedstock process flow chart

Table 1
AA6061-T651 chemical compositions

Chemical compositions (wt %)								
Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
0.67	0.52	0.28	0.02	1.10	0.19	0.01	0.02	Rest

After annealing treatment, the chips are filled in the 30 mm diameter cylindrical mold cavity and compacted by forcing the plunger that exerted on the top surface using the press machine. The process resulted in a chip-based feedstock with the target length of 25 ± 1 mm. The process was carried out at ambient temperature. The feedstock density was calculated by dividing the mass by the volume. The relative density (RD) is calculated by dividing the measured density with the as-received density of AA6061-T651, which is 2700 mm^3/Kg. Macrostructure of the chip-based feedstocks were inspected using Dynalite image capture.

The compaction experiments on the annealed chips were conducted according to 2^3 full factorial design with two replications. Table 2 shows the parameters and levels investigated in the experiment. Minitab 17 was used to generate the factorial design matrix and random order to minimize the chance of biased result. The factorial design was analysed by using analysis of variance (ANOVA). A confidence level of 5 % was chosen as the comparative value. In this analysis, calculated probability values (P-value) were compared with confidence level to identify the annealing parameter that statistically significant affecting the density of the chip-based feedstock. Compaction on the non-annealed chips were also performed for comparison.

Table 2
Parameters and levels investigated in the experiment

Parameter	Coded variable	Level		
		Low (-1)	Standard (0)	High (+1)
Annealing temperature (^{0}C)	A	300	400	500
Annealing time (min.)	B	10	35	60
Compaction pressure (bar)	C	50	100	150

3. Results

Table 3 shows the design matrix, random run order and response of the experiment. Sample 11 has the highest relative density (RD) of 97.3%. While sample 6 is the lowest with a relative density of 76.3%. The min of all value is 89%.

Table 3
Matrix design and responses

Sample no	Run order	Input parameters			Response
		A (^{0}C)	B (min.)	C (bar)	Relative density (%)
1	3	300	60	150	95.7
2	7	500	60	50	84.3
3	12	300	10	50	77.4
4	2	500	10	150	97.1
5	6	300	60	150	95.7
6	4	300	10	150	93.8
7	1	500	10	50	85.3
8	14	500	10	50	84.5
9	10	500	60	150	96.9
10	8	300	10	50	76.3
11	13	500	10	150	97.3
12	15	300	60	50	83.2
13	16	300	60	50	82.9
14	11	500	60	150	96.8
15	5	500	60	50	84.4
16	9	300	10	150	93.5

ANOVA performed on the RD, as depicted in Table 4 shows that all the terms including A, B, C, linear model, 2 way and 3-way interactions resulted in P-value less than 0.01. At 99 % confidence level, the values indicate that all terms are statistically highly significant. The results imply that there is less than 1 % error of rejecting a mean of the density has no difference.

Table 4
Analysis of variance

Source	DF	Adj SS	Adj MS	F-value	P-value
Model	7	833.589	119.084	911.65	0.000
Linear	3	798.622	266.207	2037.95	0.000
A	1	49.351	49.351	377.80	0.000
B	1	13.506	13.506	103.39	0.000
C	1	735.766	735.766	5632.66	0.000
2-Way Interactions	3	30.237	10.079	77.16	0.000
A*B	1	20.931	20.931	160.23	0.000
A*C	1	5.406	5.406	41.38	0.000
B*C	1	3.901	3.901	29.86	0.001
3-Way Interactions	1	4.731	4.731	36.22	0.000
A*B*C	1	4.731	4.731	36.22	0.000
Error	8	1.045	0.131		
Total	15	834.634			

DF- degree of freedom, Adj SS – Adj. sum of square, Adj MS – Adj. mean square

The prediction model can be denoted in an uncoded unit as shown in Eq. (1). A linear mathematical model is suggested to predict the relative density. The value of R^2 (coefficient of determination), adjusted R^2 and predicted R^2 of the suggested model are 99.87 %, 99.77 % and 99.50 % respectively. The values indicate that more than 99 % of the response variation is explained by the input variable.

$$RD = 48.67 + 0.06043A + 0.4333B + 0.2569C \quad (1)$$

where the relative density, RD, annealing temperature, A, annealing time, B, and compaction pressure, C.

Pareto chart developed from the ANOVA (as shown in Figure 2) reveals that all terms are significant since the value of standardized effect is more than 2.31. In rank order, parameters coded as C is the most significant factor followed by A, interaction AB and B. Other terms such as AC, ABC and BC approximately show levelled standardize effect.

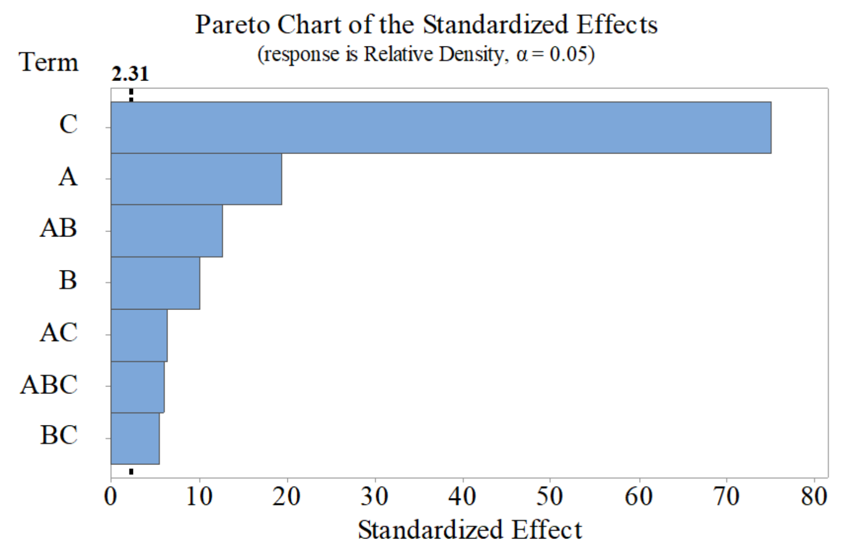

Fig. 2. Pareto chart of the standardized effect

Figure 3(a) shows a matrix interaction plot for relative density. The interaction of annealing temperature and annealing time significantly affecting the response. Factor setting at low temperature of 300 ^{0}C and low annealing time of 10 minutes result in lowest relative density. But, at the same annealing time, the relative density is increased when the annealing temperature is set at 500 ^{0}C. At the low annealing temperature of 300 ^{0}C and high level annealing time of 60 minutes, the relative density is drastically increased. However, the relative density slightly rises when the annealing temperature set at high level (500 ^{0}C). The vast difference on the slope makes the line intersect, which indicates that the annealing temperature and annealing time has significant interaction effect. The effect of all individual factors on relative density are presented in Figure 3(b). In general, response mean of all parameters is not the same across all factor level which indicates that all the factors are significant. Compaction pressure has the steeper slope followed by annealing temperature and annealing time. The steeper the slope of the line, the greater the magnitude of the main effect.

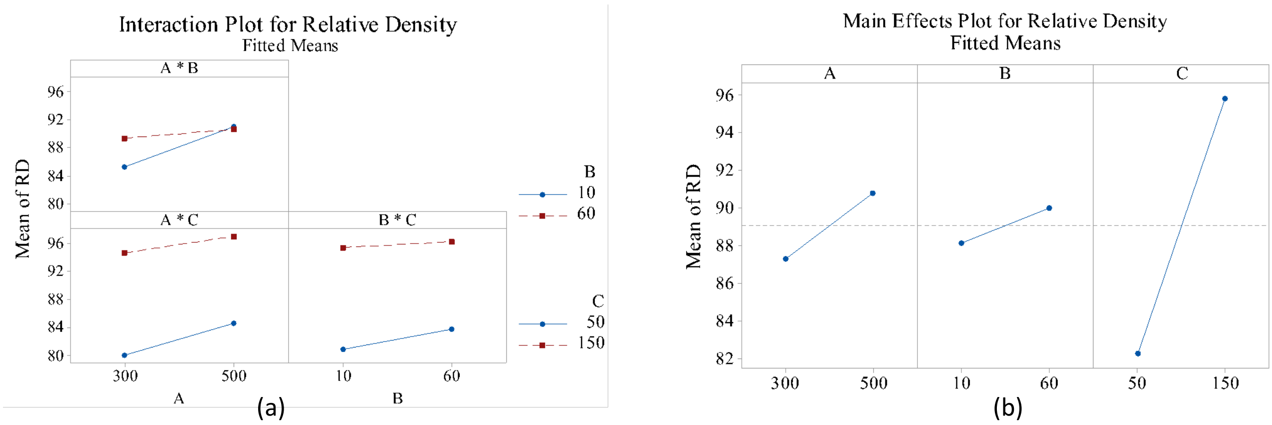

Fig. 3. Fitted means plot of relative density (a) Interaction plot (b) Interaction effect

Chip-based feedstock made from two different types of chips has resulted in different relative density as shown in Figure 4(a). The feedstock that produced from the chip without annealing and compacted at 50 bar has resulted in a relative density of 73.3 %. The relative density is rose to 85.3% when the feedstock is made from the chips that have been treated by annealing at 500 ^{0}C for 10 minutes. Chips compacted at 150 bar also shows the same pattern. The feedstock that made from non-annealed chips has a relative density of 88.3 %. The relative density improved by 9 % when the feedstock is made from annealed chip.

Visual observation on the chip-based feedstock found that the voids appear on the sample surface as shown in Figure 4(b). The feedstock with a relative density of 88.3 % exhibited more voids compared with the feedstock that has 97.1 % relative density. Distribution of the void is also inhomogeneous along the vertical. The void is more apparent on the bottom and vague as the distance toward the top. The pattern shows that the top area is denser than the bottom area.

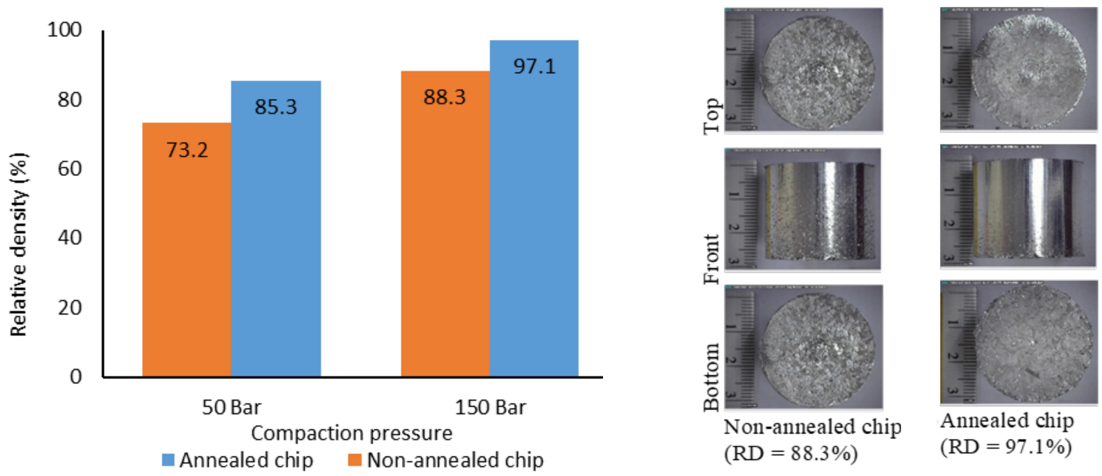

Fig. 4. Effect compaction pressure and chips (a) Relative density (b) Visual observation

4. Discussion

Annealed chips result in higher feedstock density. For example, annealed chip compacted at 150 MPa resulted in a relative density of 97.3 %. The value is 9 % higher than the chip without annealed that compacted at the same pressure. The density is increased due to the annealing has softened the chip. Annealing chip by heating above the solvus temperature of about 300 ^{0}C – 500 ^{0}C has recrystallized the structure and produce a homogeneous solid solution, allowing dissolution second phase and eliminate the segregation of the alloys. The hot chip that was left in the air rapidly cool the chip. It was because the chip has the enormous surface area to volume ratio which permits faster heat dissipation. The rapid cool limit the atom diffusion process toward potential nucleation. Thus enable the formation of super saturated solid solution of aluminium. The formation of this phase may reduce the yield stress to about 55 MPa. The softened chip enables large plastic deformation, fragmenting the thin film of aluminium oxide on the surface and thus providing larger contact surface area within the virgin aluminium. The condition permits more adjacent chip consolidated among them.

The annealing process performed on the chips has erases the effect of cold work histories such as strain hardening, residual stress and fine grain structure. A new large grain and homogenized were developed and providing the chip with high ductility [9]. In this work, annealing at a high level of 500 ^{0}C produced the highest density. This is because the chip treated at high temperature level produced grain size bigger than the low level [22]. The bigger grain size decreases the strength of the chip, reducing the spring back, increase ductility and elongation and thus increase the consolidation among the chip and density.

Annealing the chip at longer and higher temperature will be affecting the energy cost. However, it can be reduced by manipulating the temperature and time. As found in this work, chips heated with a high temperature at low and high annealing time resulted in likely similar relative density. The reason is that chip has a bigger surface area to volume ratio if compared with bulk material. It governs the physical behaviour of cooling and heating [23]. Therefore, the time required by the chip to attain the recrystallization temperature setting is less than the bulk. All alloying element is dissolved in a solid solution at a shorter time [24]. Therefore, heating the chip for a longer time such as 60 minutes, like annealing the bulk material, is unnecessary. In this work, 10 minute is enough to recrystallize the microstructure of the chip and attain large grain size.

The voids that appear on the surface of the chip-based feedstock is unfilled space between the adjunct chip boundaries. It is also representing the void pattern inside the billet. The voids are formed due to insufficient pressure and thereby the plastic flow of the aluminium chip unable to fill in space [25]. The pressure drops along the vertical also resulting voids distribution inhomogeneity. The voids distribution inhomogeneity was form due to pressure losses. The longer the distance from the plunger the lower the pressure. The voids can be eliminated by increasing the compaction pressure. However, at the higher relative density, a further increase in compaction pressure resulted in a negligible increase in relative density [19].

5. Conclusions

Annealing treatment performed on chips improved the relative density of the chip-based feedstock. The main factors affecting the relative density of the chip-based feedstock in rank order are compaction pressure, annealing temperature, interaction of annealing temperature and time, and annealing time. Longer annealing time is not required when annealing chip at high temperature level (500 ^{0}C). Shorter time is preferred since both treatments produce chip-based feedstock that the relative density has not significantly different. However, treatment at low temperature (300 ^{0}C) required longer annealing time than the treatment at high temperature.

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