Effect of Thermally-Treated Chips on Density of AlMgSi Alloys Recycled Using Solid-State Technique

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Keywords: density, direct hot extrusion, thermally formed alumina, thermally-treated chip

Abstract:

> Solid-state recycling is a sustainable technique for recycling aluminium scrap, and the process before recycling is essential to control the physical properties of the product. In this work, the effect of the thermally-treated chips on the extrudate density was investigated. The aluminium chips were thermally-treated to enrich the alumina layer and reduce compaction pressure during chips compaction before recycled using direct hot extrusion. The chips that were transformed into compacted billets were extruded directly without melting and conducted according to 24 full factorial experimental design. The density test on the recycle extrudate found that the density variation ranged from 2724 to 2983 kg/m3. The ANOVA result showed that all factors investigated were statistically significant. The most significant factor was the preheating temperature, followed by extrusion ratio, chip treatment temperature, chip treatment time, and the interaction of chip treatment-time?extrusion ratio. The predictive model suggested by the ANOVA is useful to predict the density with 1% error. Microstructure examination revealed the presence of alumina entrapped in the recycle extrudate, in which thermal-treated chips contained more alumina than that of the untreated chips. The result indicated that the thermal treatment performed on the chips had enriched the in-situ alumina, affecting the density of the recycle extrudate.

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Abstract: Solid-state recycling is a sustainable technique for recycling aluminium scrap, and the process before recycling is essential to control the physical properties of the product. In this work, the effect of the thermally-treated chips on the extrudate density was investigated. The aluminium chips were thermally-treated to enrich the alumina layer and reduce compaction pressure during chips compaction before recycled using direct hot extrusion. The chips that were transformed into compacted billets were extruded directly without melting and conducted according to 2^4 full factorial experimental design. The density test on the recycle extrudate found that the density variation ranged from 2724 to 2983 kg/m^3. The ANOVA result showed that all factors investigated were statistically significant. The most significant factor was the preheating temperature, followed by extrusion ratio, chip treatment temperature, chip treatment time, and the interaction of chip treatment-time–extrusion ratio. The predictive model suggested by the ANOVA is useful to predict the density with 1% error. Microstructure examination revealed the presence of alumina entrapped in the recycle extrudate, in which thermal-treated chips contained more alumina than that of the untreated chips. The result indicated that the thermal treatment performed on the chips had enriched the in-situ alumina, affecting the density of the recycle extrudate.

Keywords: thermally-treated chip; thermally formed alumina; direct hot extrusion; density

1. Introduction

Business competitiveness and awareness of global warming have attracted many researchers to explore the concept of sustainable manufacturing. The idea promotes minimization or elimination of waste in its production [1]. Notably, many manufacturing processes applied this concept, including recycling minute aluminium scrap via the solid-state recycling technique—the technique used to recycle aluminium chip into a semi-finished product without a melting and casting process. Literary sources [2–10] explain the details. The technique functions in less processing stages compared to the conventional technique, thus shortened cycle time, less workforce, less energy, fewer costs and less greenhouse gas emissions.

The solid-state technique uses severe plastic deformation to bond the chips together. Many types of deformation processes, including forging [4,8,11–13], rolling [14–16], and hot extrusion [17–19] were investigated in previous studies, which highlighted that the processes were feasible for the conversion of the recycled chips into a semi-finished product. Furthermore, the mechanical properties of products were also comparable to those products produced by conventional remelting techniques [20]. Figure 1 presents the schematic comparison between solid-state recycling via hot extrusion and traditional remelting techniques.

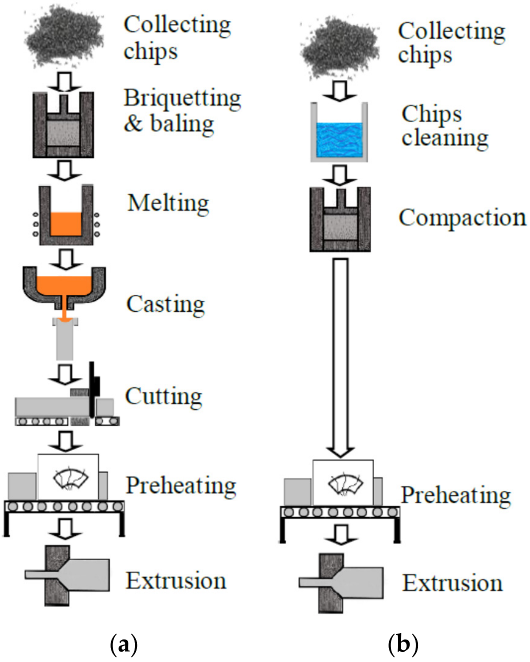


Figure 1. Aluminium recycling technique: (**a**) remelting and refine; (**b**) solid-state recycling.

Hot extrusion is typically employed to produce constant profile aluminium alloys. This process works in high temperature and pressure, which is suitable for chip weld in solid-state recycling. The process, able to severely deform the chips, shears, disrupts and fragments the in-situ alumina layer on the chip surface into ultrafine particles [21]. The fragmentation of the alumina layer eliminates the consolidation barriers, enables contact between the fresh aluminium and consolidates the chips into the bulk of extrudate. The entrapped ultrafine alumina could also improve mechanical properties trough dispersion hardening [22]. The alumina reinforces the aluminium matrix and leads to a lattice distortion, impedes the dislocation motion and increases the mechanical properties of the extrudate.

Increasing the alumina in the matrix could enhance the mechanical properties of the extrudate. Lajis et al. [6] concluded that the addition of 2% of alumina powder into the recycling chips could significantly increase the mechanical strength, but drawbacks of this technique are present. Specifically, powder agglomeration hinders the consolidation between the chips, thus deteriorates the extrudate mechanical properties [23]. Additionally, there is no bond between the chips and alumina to limit the amount of powder attached on the surface of the chip. It is also possible for the alumina to precipitate at the bottom side of the mixed chips-alumina powder, due to small size and high relative density compared to the chips, which contributes to inhomogeneous alumina distribution.

Alternatively, the reinforcement material of alumina also could be obtained by enriching the in-situ alumina layer that formed on the chip surface itself. An alumina layer of approximately four nanometers is usually formed on the surface as a result of the reaction between aluminium and atmospheric oxygen [24]. The alumina layer also could be thermally enriched in-situ through the thermal treatment on the chips before compaction. On top of that, the treatment also softens the chips, enabling cold compaction, the process before extrusion, to be performed at a significantly lower pressure compared to untreated chips [13]. The enrichment of alumina expected to give the same effect as the addition of alumina powder due to the possibility of the hot extrusion to disperse the layer into the fine particles.

Instead of improving the mechanical properties, the alumina increment in the aluminium matrix could also affect the density of the extrudate. It is because the alumina density is 3956 kg/m^3, which was higher than the as-received aluminium alloys (2700 kg/m^3). However, the research on this impact is insufficient. Therefore, this study investigated the effects of the thermal-treated chips and extrusion parameters on the density of the extrudate. An empirical model also developed in this study to estimate the extrudate density. The model could also lead to a reasonable prediction.

2. Materials and Methods

In this study, AA6061-T650 chips were collected from the milling machining. The milling was performed using MAZAK NEXUS 410A-II CNC (Yamazaki Mazak Corporation, Minokamo, Gifu, Japan) machine with a side cut end mill, a cutting tool of 10 mm diameter, cutting speed of 345 mm per second, 1 mm feed per tooth, and 1 mm depth of cut. The machining used oil-based coolant to control the cutting temperature. Acetone cleaning in the ultrasonic bath was performed to remove the contaminant and coolant, which fouled on the surface of the chip, followed with chip drying in an oven at 100 °C for one hour to evaporate the acetone and water to produce a clean chip surface [25]. Figure 2a,b shows the loose type chip collected and distribution of average total surface area to volume ratio of the chips. The average total surface area to volume ratio was calculated using the equation proposed by Hu et al. [26].

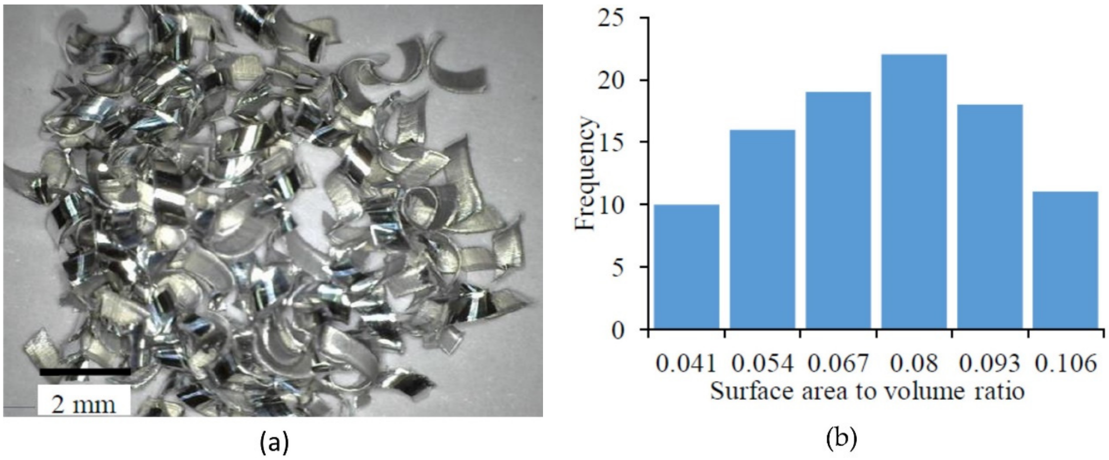


Figure 2. Aluminium chips; (**a**) chips shape; (**b**) chips total surface area to volume ratio.

The thermal treatment on chips was performed using an induction furnace. After the heat treatment, the chips were cooled off until reaching the ambient temperature. The chips were then compacted into a billet for convenient handling. The chips were filled into the compaction die cavity with 30 mm diameter and 100 mm length and compacted by the press machine. The chips were pressed with the different force to produce an equal billet density. Overall, all the compacted billets have an average height of 24 mm.

Before hot extrusion, the prepared chip-based billets were preheated at a temperature of 550 °C for one hour in the induction furnace. The billet was removed from the furnace and immediately put in the extrusion die inside the container that was heated at 350 °C. A plunger was used to push the billet through the cylindrical orifice of the flat face die using a 300-tone custom made horizontal press machine at a speed of six mm/min. Following that, the extrudate was cut off upon the completion of this process. The experiment also extruded solid as-received aluminium alloys and untreated chips for comparison using the extrusion ratio of six and preheating temperature of 550 °C. The extrudates were naturally cooled off to an ambient temperature after extrusion. Figure 3 schematically illustrates the overall process involved in the experiment.

Figure 3. Extrusion procedure in recycling aluminium chips.

The experimental research design involved four factors two levels, as shown in Table 1, and the response variable was the extrudate density. The experiment was performed based on 2^4 full factorial design with two replications. The design also included three centre points to determine the curvature effects of the model. Minitab 17 was employed to generate the design matrix and the random run order to eliminate the probability of the biased result. Overall, the experiment involved a thirty-five runs order. The analysis of variance (ANOVA) performed on the result determined the effect of the parameters on the extrudate density. A calculated probability value (*p*-value) of equal to 5% or lower indicated the rejection of the null hypothesis and the statistical significance of the factor.

Table 1. Factors and levels.

Factors	Factors Symbol	Levels		
		Low	Centre	High
Chip treatment temperature (°C)	A	300	400	500
Chip treatment time (minute)	B	10	35	60
Extrusion ratio	C	6	9	12
Preheating temperature (°C)	D	300	425	550

Without additional heat treatment, the extrudates were cut into a sample using a metallographic cutting machine. The density measurement conducted on a small piece of the extrudate was performed according to Archimedes principles using the density balance. Samples for microstructure examination were cut parallel to the extrusion direction. The samples were ground by alumina sandpaper, polished by diamond paste on polishing cloth, etched by a Baker reagent, and examined by Olympus BX60M optical microscope (OPM) (Olympus Coporation, Nagano, Japan). The energy-dispersive X-ray spectrometer (EDS) was carried out for analyzing the chemical constitution using a HORIBA E-MAX X-ACT model 51-ADD0009 (Horiba Limited, Middlesex, NJ, USA.). Figure 4 shows the product of recycling aluminium produced in the experiment.

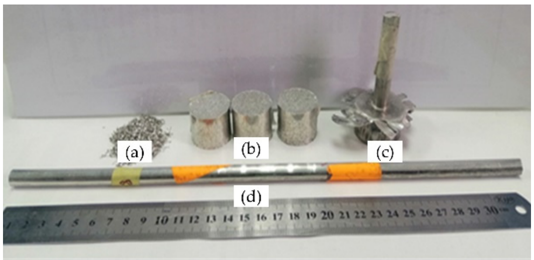


Figure 4. Recycle chips conversion; (**a**) aluminium chips; (**b**) compacted billet; (**c**) discarded extrudate; (**d**) extrudate billet.

3. Results

Table 2 shows the design matrix and extrudate density response of thermally-treated chips performed as per the experimental plan. It was found that the density variation of the recycle extrudate ranged from 2683 to 2724 kg/m^3. Meanwhile, extrusion performed on solid as-received and untreated chips resulted in a density of 2702 and 2678 kg/m^3 respectively The extrudate of thermally-treated chips that produced with all factors set at a high level resulted in the greatest density of 2724 kg/m^3. This value is 25 kg/m^3 more than the extrudate of solid as-received. In contrast, the extrudate produced with all factors set at a low level resulted in the lowest density of 2683 kg/m^3. All the extrudate made of thermal-treated chips has higher density compared to untreated chips, but the density of as-received is within the range of treated chips.

Table 2. Experimental design and the response.

Sample	Run Order	A (°C)	B (min)	C	D (°C)	Density (kg/m^3)
1	9	300	10	6	300	2.683
2	20	500	10	6	300	2.688
3	32	300	60	6	300	2.689
4	34	500	60	6	300	2.699
5	27	300	10	12	300	2.694
6	35	500	10	12	300	2.707
7	28	300	60	12	300	2.701
8	4	500	60	12	300	2.711
9	16	300	10	6	550	2.703
10	7	500	10	6	550	2.702
11	6	300	60	6	550	2.706
12	24	500	60	6	550	2.710
13	1	300	10	12	550	2.705
14	11	500	10	12	550	2.721
15	30	300	60	12	550	2.719
16	10	500	60	12	550	2.724
17	2	300	10	6	300	2.687
18	5	500	10	6	300	2.698
19	22	300	60	6	300	2.698
20	31	500	60	6	300	2.707
21	12	300	10	12	300	2.687
22	33	500	10	12	300	2.707
23	26	300	60	12	300	2.705
24	15	500	60	12	300	2.713
25	8	300	10	6	550	2.702
26	3	500	10	6	550	2.708
27	13	300	60	6	550	2.706
28	17	500	60	6	550	2.703
29	21	300	10	12	550	2.705
30	29	500	10	12	550	2.717
31	25	300	60	12	550	2.708
32	23	500	60	12	550	2.719
33	18	400	35	9	425	2.705
34	14	400	35	9	425	2.708
35	19	400	35	9	425	2.707

Table 3 shows the ANOVA results after backward elimination. The main effects A, B, C, D, and AC interaction has a *p*-value less than 5%, which indicated that the terms were statistically significant. The Pareto plot of effect in Figure 5a shows that factor D results in the largest effect on the density as it extends the farthest, which was followed by C, A, and B. The effect of AC interaction was the least significant since it extends the least. Others interaction terms did not reach statistical significance and were discarded from the analysis. Meanwhile, the curvature and lack of fit also were not significant, which indicates that a linear model was sufficient enough to fit the response. The impact of the interaction between factors A and C could be explained based on the interaction effect plot, as shown in Figure 5b. The extrusion performed with both factor A and B set at high levels gives the most significant effect on density compared to factor A or B alone. The main effect plot illustrated in Figure 5c shows that all factors had a positive effect on the density mean. An increase in the factor levels would increase the density. It also could be seen from the comparison between the slopes of each main effect that no significant difference was present between the magnitude of the effect. Notably, the preheating temperature gives the most notable impact followed by extrusion ratio, chip treatment temperature, and chip treatment time.

Table 3. ANOVA results after backward elimination.

Source	Degree of Freedom	Adj. Sum of Square	Adj. Mean of Square	F-Value	*p*-Value
Model	5	0.002314	0.000463	25.40	0.000
Linear	4	0.002230	0.000557	30.59	0.000
A	1	0.000435	0.000435	23.88	0.000
B	1	0.000364	0.000364	20.00	0.000
C	1	0.000630	0.000630	34.58	0.000
D	1	0.000800	0.000800	43.90	0.000
AC	1	0.000084	0.000084	4.64	0.040
Curvature	1	0.000049	0.000049	2.83	0.103
Lack-of-fit	10	0.000089	0.000009	0.41	0.923
Pure error	18	0.000391	0.000022		
Total	34	0.002843			

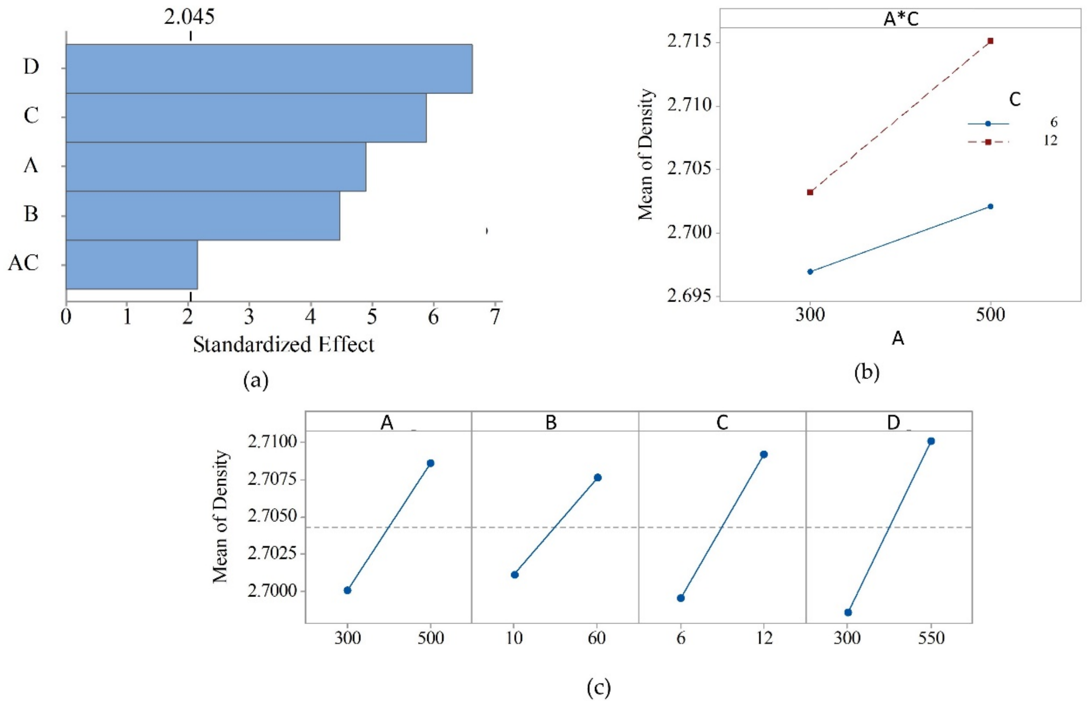


Figure 5. ANOVA results; (**a**) standardized effect; (**b**) interaction effect between chip treatment temperature and extrusion ratio; (**c**) main effect plot.

The linear regression model derived from the ANOVA results shows a clear relationship between the independent variable and the response. The model has determination coefficient R^2, adjusted R^2, and predicted R^2 of 81.4%, 78.2%, and 73.4%, respectively. The R^2 indicated that more than 81% of the variation in the response variables explained by the input variables. The difference between adjusted R^2, and predicted R^2 with no more than 20% also indicated that the model was not overfitted. Equation (1) shows the model in an uncoded unit, which is beneficial for predicting the response within the design space by giving the input value of the factor.

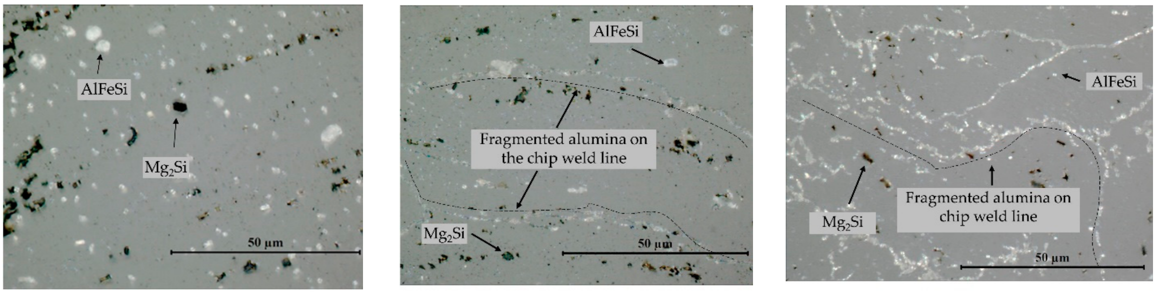
$$\text{Density} = 2.6742 - 0.000012A + 0.000135B - 0.00069C + 0.00004D + 0.000005AC \quad (1)$$

Table 4 shows the result of three additional experiments performed to validate the predictive model. It was found that the error between the prediction model and the experimental measurement did not exceed 1%. The error demonstrated that the developed prediction model was accurate and reliable to estimate the density of the extrudate made of thermally treated chips.

Table 4. Model validation experiment.

Test	Factor Condition				Predicted Density (kg/m^3)	Measured Density (kg/m^3)	Error (%)
	A (°C)	B (Min)	C	D (°C)			
1	300	35	6	300	2.692	2.678	−0.5%
2	500	60	12	425	2.715	2.725	0.4%
3	400	35	9	425	2.703	2.703	−0.4%

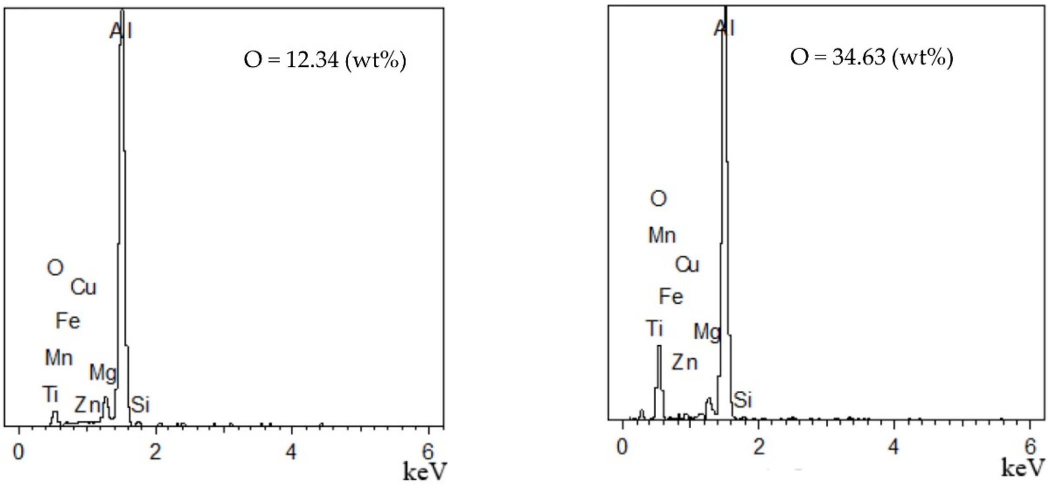
Figure 6 compares the microstructure of extrudate made from solid as-received, untreated chip, and the 500 °C thermal-treated chip. Overall, all the extrudates exhibited the presence of Mg_2Si and spheroidal Al-Fe-Si rich precipitates, which could be identified from the black spot and light grey, respectively. This result was similar to the finding in the study by Voort et al. [27]. The extrudate of solid as-received consist of coarse Mg_2Si and AlFeSi precipitate, while the chip-based extrudates of untreated and thermally-treated showed fine precipitate. The chip weld lines were clearly seen on the untreated and thermally-treated extrudate but not in the solid as-received extrudate. The weld lines formed due to chips consolidation. No micropore and crack found in the microstructure indicated the process produced good weld strength between the chips. Fragmented alumina as seen as a white contrast was clearly visible entrapped in the aluminium matrix of both chip-based extrudates, the untreated and thermally-treated. The alumina in untreated mainly existed in the chip weld line. However, the presence of alumina in the thermally-treated appeared to be more apparent compared to the untreated. The alumina not only appeared in the weld line but was also found scattered on the entire microstructure.



(a) (b) (c)

Figure 6. Microstructure of the extrudate; (**a**) as-received; (**b**) untreated chip; (**c**) 500 °C thermally-treated chip.

EDS spot analysis performed on the white contrast on the chip weld line showed the presence of elements such as aluminium (Al), oxygen (O), copper (Cu), Magnesium (Mg) and silicon (Si) and manganese (Mn) as high-intensity peaks, as shown in Figure 7a,b. High peaks of oxygen proved the white contrast area mainly contains alumina substances. The extrudate of the untreated chip contains 12.34% (in weight) oxygen. The oxygen content in extrudate made of 500 °C thermally-treated chips recorded a higher oxygen content of 34.63%, which was an increment of 22.3% compared to untreated chips. The increment proved that the thermal treatment performed on the chips had enriched the alumina formation.



(a)

(b)

Figure 7. Energy-dispersive X-ray spectrometer (EDS) result; (**a**) untreated chip; (**b**) 500 °C thermally-treated chip.

4. Discussion

The variation in the extrudate density of thermally-treated chips was due to the presence of alumina in the aluminium matrix and the porosity. During extrusion, the plunger pushed the compacted chip through the die cylindrical orifice. Notably, the difference in the diameter of the compacted billet and the orifice resulted in high shear stress and severely deformed the compacted chips. Furthermore, the alumina layer, which was naturally or thermally formed on the chip surface, was heavily fragmented into fine particles and entrapped in the aluminium matrix. On the other hand, low shear stress could result in unbroken alumina layer, which prevented a good consolidation between the chips. Subsequently, porosity could be formed between the chips, which might lead to a more severe case, in which the chips could slide along each other rather than being deformed and consolidated [28].

It was demonstrated in the ANOVA result that the presence of alumina and the porosity formed in the extrudate was influenced by the chip treatment temperature, chip treatment time, extrusion ratio, and preheat temperature. The first two factors, namely chip treatment temperature and chip treatment time, influenced the thickness of the alumina layer [29], while the last two factors, namely extrusion ratio and preheat temperature, influenced the shear strain during the extrusion. Notably, while the increase in the extrusion ratio led to a further shear strain on the chips during the extrusion, the rise in the chip treatment temperature and time could enhance the thickness layer of in-situ alumina on the chip surface, which increased the density further. Furthermore, the high preheating temperature would soften the chips, leading to further shear strain. In solid-state recycling of aluminium chip, high shear strain is normally required for the fragmentation of the alumina layer to produce a good consolidation, eliminate the porosity and increase the density.

The interaction of factor A and C indicated that the extrusion performed at high extrusion ratio and the chips treated at high treatment temperature resulted in a more significant effect on density. This could be explained by the fact that chips treated at high temperature led to enriching the in-situ alumina more than the treatment performed at low temperature [29]. The higher alumina formed on the chip surface resulted in more in-situ alumina entrapped in the extrudate. Any small amount of alumina entrapped in the extrudate would significantly affect the density of extrudate as the density of alumina is higher than the aluminium by a factor of two. Meanwhile, the extrusion performed at a high extrusion ratio eliminated the micropore between the consolidated chips and improved the density. Therefore, the density of the extrudate made from aluminium chips could be higher than as-received aluminium alloy. In the case of low-level extrusion ratio, the shear strain developed during the extrusion might not be sufficient to break the hard alumina layer into a fine particle, producing more micropores, and resulting in a lower density.

The extrudates made of the thermally-treated chips at higher temperature were found to have higher extrudate density compared to the chip treated at a lower temperature. This finding explained the effect of the in-situ alumina enrichment due to thermal treatment. According to Hunter and Fowle [29], the thickness of the alumina layer formed on the aluminium surface treated at a higher temperature was higher compared to the chip treated at a lower temperature. The thickness of the alumina layer increased with the rise in the thermal temperature and thermal treatment time. This was due to the fact that the activation energy at a higher temperature improved oxygen diffusion in the alumina barrier, which allowed further chemical reaction to occur between the aluminium and oxygen and further enrichment of alumina. It was also proven in the microstructure examination that the presence of the in-situ alumina was more prone compared to the untreated chips.

The extrudate made from the thermal-treated recycle chip was found to have a density as high as 2724 kg/m^3 (sample 16), which was 0.7% higher compared to the as-received material. Provided that the chips were cleaned by an ultrasonic cleaner in acetone bath and dried in the oven, it was believed that the density of recycling chips extrudate was higher compared to the as-received material was solely due to the vast difference between the density of the alumina and aluminium alloys. Theoretically, the alumina had a density of 3950 kg/m^3, which was higher than the original aluminium alloys. Therefore, the presence of a small amount of alumina in the matrix could significantly increase the density of the extrudate.

The model proposed in this work could be used to predict the density of the extrudate that performed within the design space of the experiment. The density was mainly attributed to chip treatment temperature, chip treatment time, extrusion ratio and preheat temperature. High chip treatment temperature and chip treatment time would enhance the alumina formation on the aluminium alloys chip surface [29]. Besides the impacts on density, it was shown that the increment of the alumina in the aluminium matrix also affected mechanical properties of the extrudate due to dispersion hardening. As found by Chiba et al. [14], Wagiman et al. [30] and Azlan et al. [31], the extrudate made of recycling chip exhibited higher strength properties compared to the as-received material.

5. Conclusions

Direct hot extrusion is an alternative technique in recycling aluminium chips. In this work, the thermal-treatment performed on the chips prior hot extrusion was found to significantly affect the density of the extrudate due to alumina enrichment on the surface of the chip. The ANOVA result indicated that chip treatment temperature, chip treatment time, extrusion ratio, preheating temperature, and interaction between chip treatment temperature and extrusion ratio statistically significantly affected the density. The density of the extrudate can be estimated using the linear prediction model that has been developed with an error of less than 1%. Comparison between the microstructure of the extrudate proved that thermal-treated chips contained more alumina compared to the untreated chips and as-received. The result indicated that the process before hot extrusion plays an important role in the physical properties of the extrudate.

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