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EFFECT OF FLUX APPLICATION DURING IN-SITU CASTING AS A DIRECT RECYCLING OF ALSI7MG ALUMINIUM ALLOY MACHINING CHIPS

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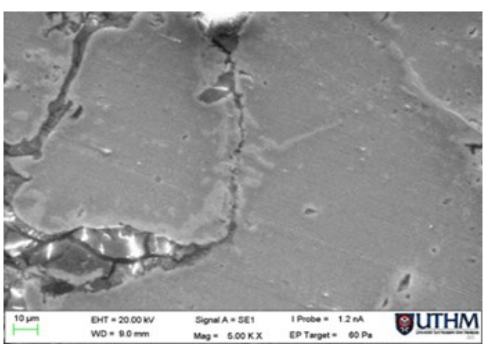
Graphical abstract



Abstract

Recycling aluminium (Al) waste by secondary smelting production saves 95% energy had encourages a new alternative recycling technique known as the in-situ casting or melting to transform waste into product directly. Therefore, aluminium (AlSi7Mg) machining chips with flux addition were heated for 30 minutes in a laboratory furnace at 650°C and 700°C. The physical of Al chips turned from sparkling silvery grey to dusty grey after being heated indicating oxidation. The machining chips had partially melted and fused together however being interrupted by thin oxide layer. Scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) reveal the Al and oxygen (O), while x-ray diffraction (XRD) analysis confirmed aluminium oxide (Al_2O_3) present in the fused Al chips.

Keywords: Aluminium, In-situ casting, In-situ melting, Aluminium Oxide, Aluminium recycling

Abstrak

Pengitaran semula sisa Al berpotensi menjimatkan sehingga 95% tenaga. Salah satu Teknik penjimatan adalah melalui tuangan atau peleburan secara in-situ. Kaedah ini berkeupayaan menghasilkan produk terus dari pemprosesan yang dilakukan. Oleh itu, cip pemesinan aluminium (AlSi7Mg) dengan tambahan *flux* telah dipanaskan selama 30 minit di dalam relau makmal pada julat suhu 650°C dan 700°C. Keadaan fizikal cip Al bertukar daripada warna kelabu keperakan berkilauan kepada kelabu berdebu selepas dipanaskan. Ia menunjukkan cip telah mengalami pengoksidaan. Sewaktu itu, cip pemesinan telah separa cair dan bercantum bersama namun diganggu oleh lapisan oksida nipis. Mikroskop elektron pengimbasan (SEM) yang dilengkapi dengan spektroskopi penyebaran tenaga (EDS) mendedahkan kehadiran Al dan oksigen (O), manakala analisis pembelauan sinar-x (XRD) mengesahkan aluminium oksida (Al_2O_3) hadir dalam cip Al yang telah bercantum.

Kata kunci: Aluminium, Perleburan *In-situ*, Tuangan *In-situ*, Aluminium Oksida, Kitar Semula Aluminium

1.0 INTRODUCTION

Aluminium alloys have seen increased use recently because of their beneficial characteristics [1]. Aluminium (Al) is a metal derived from bauxite used to make various industrial goods. The bauxite mining business started with little regard for possible negative environmental consequences, economic concerns, or human well-being. Surface mining, for example, is one of the most common extraction processes involving plant removal [2]. Compared to primary aluminium production or bauxite consumption, secondary aluminium manufacturing or recycling uses around 10 to 15 times less energy [3].

Recycling aluminium offers environmental benefits that extend beyond energy conservation and emission reduction. In addition to these advantages, aluminium recycling contributes to water conservation by utilizing significantly less water than the initial manufacturing process. Furthermore, recycling decreases trash creation and the demand for landfill space since aluminium may be collected, processed, and turned into new objects rather than wasted [4].

Aluminium waste, such as swarf or chips, is increasing in volume. The recycling of aluminium alloy chips has developed as a critical problem and a topic of academic inquiry due to a rising awareness of environmental sustainability [5]. The best type of waste to remelt for reuse is aluminium chips, which are formed during machining. The elevated ratio of surface area to volume in aluminium chips intensifies the prominence of oxidation. Chips are unsuited for traditional recycling techniques due to their spiral and elongated structure [6].

Currently, aluminium chip waste is recycled by the scrap recycling industry. However, the traditional recycling process has many steps. The chips need to compact into a briquette, and then the briquette will go through a melting or refining process, casting, and the process continues until the forming process of the end product [7]. The benefits of aluminium's direct recycling are significant since they may reduce costs for environmental protection and provide better energy utilisation [8]. The investment casting method has much potential for the direct recycling of aluminium waste. The conventional investment casting process, on the other hand, has several disadvantages [9]. Entrained air bubbles and liquid metal oxide coatings are two prominent flaws caused by metal pouring turbulence during the casting process. The oxides are always entrained, and a bifilm creates casting fractures [10]. During the pouring process, the aluminium melt forms a thin oxide covering within and aluminium oxide (Al_2O_3), which folds due to turbulence and nucleated porosity [11]. Oxide inclusions (Al_2O_3) are inevitably generated and imprisoned inside the melt during the melting of aluminium. Without appropriate molten aluminium refining methods, these oxide inclusions will persist in the aluminium post-casting, leading to various challenges, including diminished mechanical properties, compromised surface quality and poor machinability [12].

In-situ casting can be an alternative to the pouring-free casting process. The in-situ casting of metal alloy material, such as magnesium alloy granules, can produce outstanding results. When granules and flux powder are mixed in a certain amount by weight and heated at 650°C for 30 min in a heating furnace, the granules might melt completely [13]. The salt flux, composed of equimolar NaCl-KCl and featuring a low melting point of approximately 660°C, is a frequently employed element in the recovery of aluminium from waste aluminium and aluminium dross. This flux envelops the liquid aluminium, inhibiting additional oxidation, removing the oxide layer, and enhancing the coalescence of aluminium droplets [14]. The salt flux used for melting aluminium alloy chips functions as a covering agent. Cover fluxes provide a protective layer on the surface, functioning as a diffusion barrier to prevent oxygen from interacting with the melt. Cover fluxes are often made up of inorganic salts liquid at melting temperatures. Salt fluxes for covering, refining, degassing, and drossing often use NaCl-KCl mixes due to their low melting temperature and minimal reactivity with the melt [15].

In addition, a novel approach involves in-situ microwave casting for metallic materials, providing an alternative method for casting metal objects. The charge's electromagnetic and thermal properties affect how the microwave interacts with the material and melts. A dense cast was made with less than 2% porosity. However, it is an expensive and complicated procedure [16].

In this research, we have used the aluminium alloy AlSi7Mg machining chips for a direct recycling process. The heating temperature used in this research must be approached with the Al-Si eutectic temperature. Based on the Al-Si phase diagram, the eutectic reaction occurs at 12.6 wt. % Si and 577°C. The aluminium alloy chips used in this experiment have 7 wt. % of silicon, which shows that the melting temperature is around 620°C [17].

Heating in an argon atmosphere was unable to completely prevent the aluminium granules from oxidising during heating. A similar result was observed when the aluminium granules were heated at 850°C for 30 min in an argon atmosphere compared to those in air [18].

Besides that, alternative methods must be implemented to reduce costs and enhance the porosity of the investment-cast aluminium alloy. Therefore, in-situ casting was developed using a standard laboratory furnace with flux application. The influence of flux application on casting porosity and mechanical performance will be investigated throughout the development of the pouring-free casting process by in-situ casting method for aluminium alloy machining chips.

2.0 METHODOLOGY

2.1 The Preparation of the Ceramic Mould

A 34mm-by-65mm cylindrical mould was used to make the pattern for investment casting ceramic mould. The pattern was dipped in a ceramic slurry made from 75-micron Zircon ($ZrO_2.SiO_2$) flour and colloidal silica (SiO_2). The dipped slurried pattern undergoes drying for an hour for face coating purposes. Subsequently, a second coating was administered through a repeated immersion in the slurry, promptly followed by the application of a fine-grain (0.3-0.7mm) alumino-silicate ($Al_2O_3.SiO_2$) layer, serving as a supplementary coat. Following an hour of drying, the coated pattern underwent another round of immersion in the slurry, this time coupled with the application of a coarser (0.7-1.0mm) alumino-silicate ($Al_2O_3.SiO_2$) sand, repeated six times. The pattern was immersed in the slurry for the final coat and dried for 24 hours. The stuccoed pattern was subsequently subjected to a one-hour dewaxing process in a furnace at 200°C, resulting in the ceramic mould's wall thickness ranging from 6 to 7mm.

2.2 The Heating Process of Aluminium Alloy (Al-Si7Mg) Chips

1 mm width aluminium alloy (Al-Si7Mg) chips, with lengths ranging from 5 to 7mm and obtained from machining waste, were put into the ceramic investment casting mould at a quantity of 20g. The ceramic mould, housing the aluminium alloy chips, was then positioned within the laboratory furnace, model Protherm Furnace PLF 120/7, for an in-situ casting procedure. The aluminium alloy chips underwent a 30-minute heating process at 650°C and 700°C, as indicated in Table 1. Two sets of aluminium chip samples were heated without flux, while another two sets was heated in the presence of flux salt. The flux used is 50wt% sodium chloride (NaCl) and 50wt% potassium chloride (KCl), and the flux ratio used is 1:0.1(2g), and the method for applying flux is to mix all of the flux with the chips before heating. After heating, the aluminium alloy samples were cooled to room temperature and then prepared for metallographic analysis.

Table 1 Temperature and flux weight for aluminium alloy chips

No.	Heating Temperature (°C)	Flux Weight (g)
1	650	0
2	650	2
3	700	0
4	700	2

2.3 Physical Evaluation, Oxidation Analysis and Thermal Analysis

The compounds and phases present in the oxide layer of aluminium alloy chips samples during in-situ melting was examined using visual methods for physical observation, scanning electron microscope (SEM) with Energy Dispersive Spectroscopy (EDS) model EVO LS10 to analyse the oxide structure, surface morphology, and elemental analysis, X-ray diffraction (XRD) model Bruker D8 for oxide compounds and phases analysis and Thermogravimetry Analysis (TGA) model TA Instruments TGA Q500 for thermal analysis.

3.0 RESULTS AND DISCUSSION

3.1 Physical Evaluation

Figures 1(a-b) show pictures of aluminium alloy chips without flux. Figure 1(c-d) depicts images of aluminium alloy chips mixed with flux after being heated for 30 minutes at two different temperatures. Both samples with and without flux heated at 650°C and 700°C, depicting partially melting chips that unable to develop a molten aluminium pool. Upon visual inspection, it was observed that the colour of the aluminium alloy chips changed after being subjected to temperatures exceeding the melting point of 610°C [19]. Remarkably, the chips retained their original geometry. This phenomenon may be attributed to the active surface oxidation occurring during the in-situ casting of aluminium alloy chips. This process results in a thin oxide layer on the surface, impeding the melting of smaller-sized aluminium particles [11]. The heated aluminium alloy chips without flux application are unable to fuse. The main difference between the heated and unheated aluminium alloy chips was their silvery glossy colour, which turned to dusty grey once heated during the in-situ casting experiment. When a metal is subjected to a heating procedure, the final results may differ amongst samples heated to different temperatures [20]. Figures 1(c) and 1(d) depict the agglomeration of the aluminium alloy chips that experienced in-situ casting with flux at 650°C and 700°C. The agglomeration of aluminium alloy chips indicated that applying flux with specified ratios increases, promoting the partial melting of the aluminium alloy chips during the in-situ casting experiment. The participation of the flux in adhering oxide inclusions from the molten metal to the surface facilitated the agglomeration of the chips [21]. Furthermore, the flux endeavoured to disrupt the formation of surface oxide on the aluminium alloy chips, thereby encouraging the fusion of the chips during the in-situ casting process which is agree with other researchers [11].

Figure 1 Heated aluminium chips (a) without flux at temperature 650°C; (b) without flux at temperature 700°C; (c) with flux at temperature 650°C; (d) with flux at temperature 700°C

3.2 Oxide Structure, Surface Morphology and Elemental Analysis

Figure 2 shows SEM surface morphology and cross-section images of the oxides on aluminium alloy chips after being heated for 30 min at 650 and 700°C during in-situ casting experiments. Figures 2(a) and 2(c) show SEM pictures of the oxide morphology on the surface of aluminium alloy chips, depicting a wrinkled surface with a tiny oxide nodular. The oxide nodule was detected developing and growing on the surface of the chips. Individual and clustered oxide nodules were distributed randomly across the surface of the chips, giving rise to larger oxide formations. While the cross-section image shown in Figures 2(b) and 2(d) hardly shows oxide nodules due to the smaller size of the chips. However, the elemental analysis using EDS, as shown in Figures 4(a) and 4(b), unveiled the existence of oxygen on the surface, indicating that oxidation occurred during in-situ casting. The oxidation of molten aluminium results in an initial thin oxide and the quick integration of oxygen and aluminium [22].

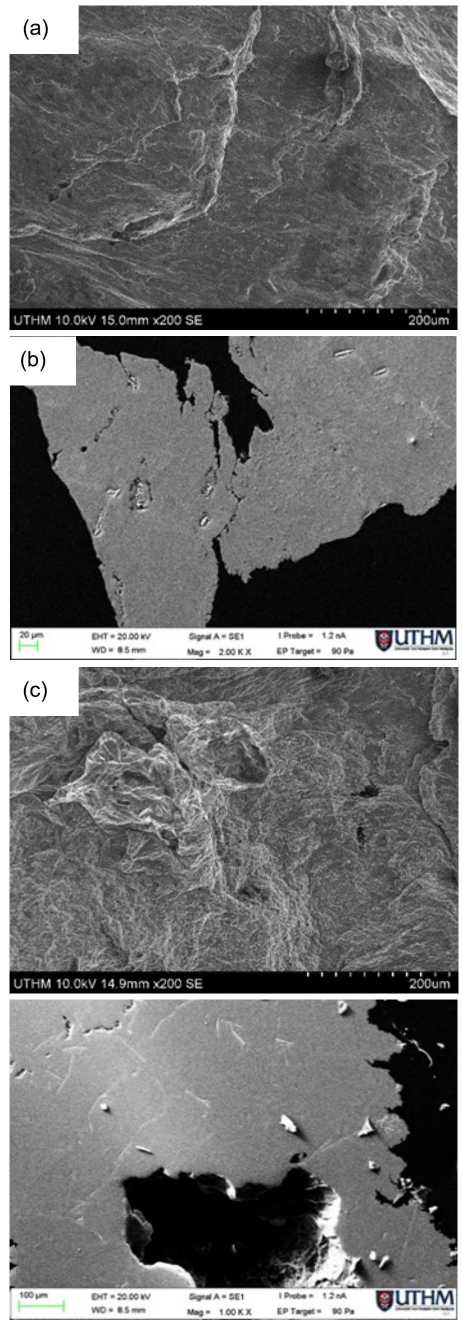


Figure 2 (a) - (c) and (b) - (d) are SEM images of the morphology of the oxide on the surface of aluminium alloy chips and cross-section images of the aluminium alloy chips, respectively, after being heated at the temperatures of 650 and 700°C for 30 min without flux

Figure 3(a) shows an SEM image of raw or unheated aluminium alloy chips. Figure 3(b)-3(e) shows SEM pictures of the surface morphology and cross-section of the oxides on aluminium alloy chips mixed with flux that had been heated for 30 min at 650°C and 700°C during in-situ casting.

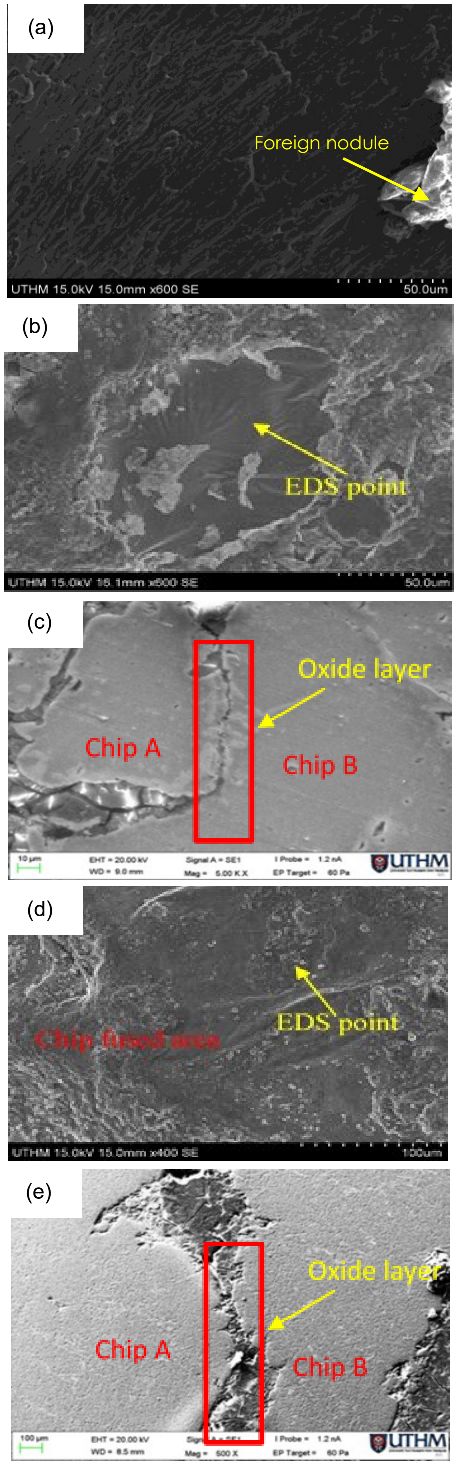


Figure 3 (a) SEM images of the morphology of the unheated aluminium alloy chips, (b) - (d) and (c) - (e) are SEM images of the morphology of the oxide on the surface of aluminium alloy chips and cross-section images of the aluminium alloy chips with flux after being heated at the temperatures of 650°C and 700°C for 30 min

Based on Figure 3(a), the microstructure image shows that in the raw chip image, the surface is entirely black in colour and clear from oxide nodules, and the structure of the rough surface is because of the machining process. Figures 3(b) and 3(d) show SEM pictures of the oxide morphology on the surface of aluminium alloy chips that fused with other chips in the ceramic investment casting mould. Figures 3(b) and 3(d) show that the black area (in the circle) indicates the melting of aluminium. It can be seen in Figures 3(b) and 3(d) that the black area on the chips was free from the oxide nodules. However, the region outside the circle exhibits individual and clustered oxide nodules resulting from the active surface oxidation during the in-situ casting process.

The images shown in Figures 3(c) and 3(e) indicate that the aluminium chip (denoted as Chip A) attempted to fuse with another chip (denoted as Chip B) in the investment casting ceramic mould. Nevertheless, the process was impeded by the oxide layer that developed on its surface during the in-situ casting process. The inability for complete melting and fusion to occur, attributed to the presence of oxygen and moisture during in-situ casting, played a role in the active oxidation of the aluminium alloy chips, as [11] reported.

The EDS analysis was conducted to ascertain the existence of the element on the surface of the aluminium alloy chips following a 30-minute heating to temperatures of 650°C and 700°C. The EDS spectrum confirmed the presence of Al and O peaks, as illustrated in Figures 4(a) and 4(b), showing that the heated chips were oxidised. Figure 4(a) shows that oxygen was 34.59wt.%, indicating that the aluminium alloy chips had oxidised during the in-situ casting experiment. The EDS spectra of the aluminium alloy chip surface heated for 30 minutes at 700°C are shown in Figure 4(b). The maxima for aluminium (Al) and oxygen (O) in the spectrum are 32.92wt.% and 30.86wt.%, respectively.

Aluminium alloy chips experienced oxidation when subjected to heat in the furnace during the in-situ casting experiment, as evidenced by the presence of oxygen in the oxide morphology of the heated sample. The surface oxidation of the chips impeded the progress of the in-situ casting experiment, hindering the release of the molten material and the formation of a molten alloy pool. This occurrence is likely linked to the active surface oxidation of the chips during the melting process, leading to the formation of a thin oxide layer within the melt [11]. Figure 4(c) shows the EDS analysis taken from the EDS point, as shown in Figure 3(b), which is the area where the chips fused with other chips. The result shows that the aluminium weight percentage is high while the oxygen weight percentage is deficient (4.5%). It shows that very low oxygen in that area allows the chip to attempt to melt and fuse with other chips in the ceramic investment casting mould. While Figure 4(d) depicts the high peaks of oxygen (58.28%) indicating the oxides were formed at the EDS point as in Figure 3(d).

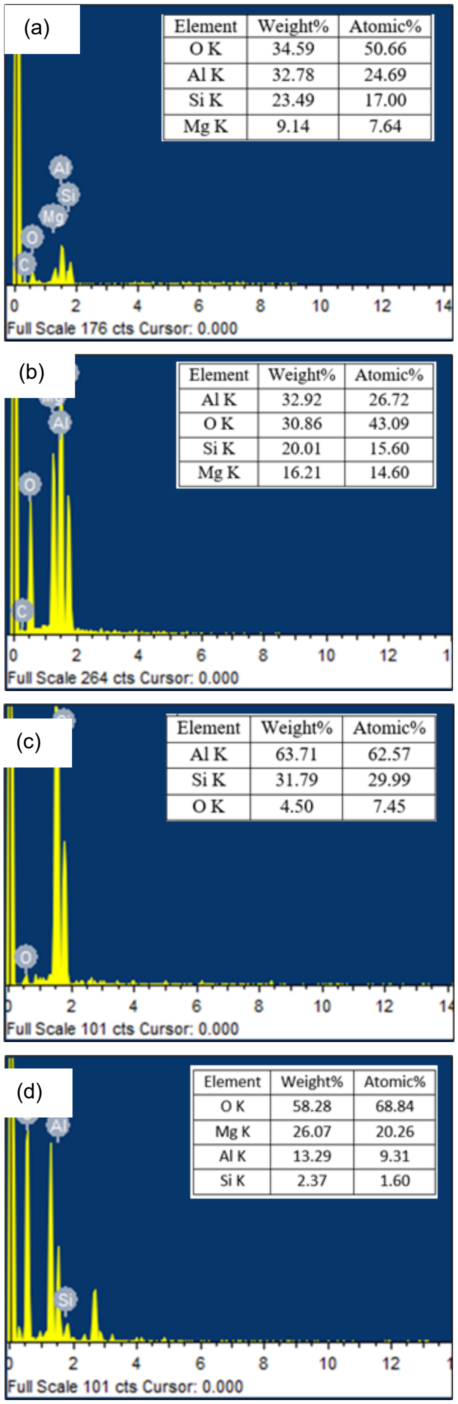


Figure 4 (a) - (b) and (c) - (d) are EDS analyses of the morphology of the oxide on the surface of aluminium alloy chips without flux and with flux, respectively, after being heated at the temperatures of 650°C and 700°C for 30 min

3.3 Oxide Compounds and Phases Analysis

Figure 5 depicts the XRD analysis of aluminium alloy chips before and after 30-minute heating at 650°C. The XRD spectrum for the aluminium alloy chip sample before heating displayed distinct crystalline aluminium peaks at 2θ degree angles of 38.47°, 44.74°, 65.14°, 78.23°, and 82.44°, as illustrated in Figure 5(a).

Aluminium oxide (Al_2O_3) has been discovered as a significant phase, as shown in Figure 5(b). The XRD pattern revealed diaspore [α-AlO(OH)], which matched the crystalline aluminium peaks for the heated samples. The hydration of aluminium caused the production of diaspore during in-situ casting at the heating temperature of 650°C. It is also shown that [γ-Al_2O_3] was detected due to the hydrated aluminium. Therefore, the transition of boehmite [γ-AlO(OH)] led to the production of metastable [γ-Al_2O_3]. The Al_2O_3 formation was due to the presence of oxygen and moisture when heating at elevated temperatures during in-situ casting experiments, as discovered by [23]. The transition of the oxide's phases from aluminium hydroxide to alumina was believed to follow the transformation phases reported by various researchers [18].

The emergence of Al_2O_3 corroborated the conclusion of this investigation, indicating that the inadequate melting and fusion of the aluminium alloy chips resulted from the oxide layer forming on their surface. This insulating oxide layer during in-situ casting played a role in impeding the process.

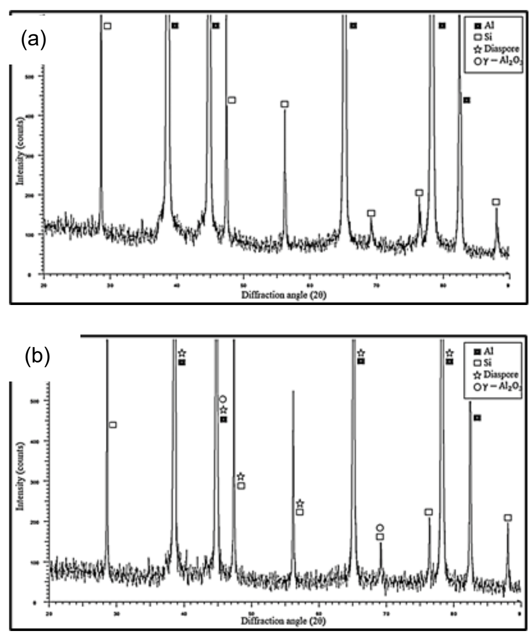


Figure 5 XRD analysis of aluminium alloy chips (a) before heating-raw aluminium machining chips (b) after 30 minutes of heating at temperatures of 650°C during the in-situ casting experiment

3.4 Thermal Analysis

Figure 6 depicts the TGA graph analysis of aluminium alloy chips starting from the room temperature to 900°C in air atmosphere. TGA-DTG analysis was carried out to investigate the thermal stability AlSi7Mg chips. The TGA-DTG curves shown in Figure 6 exhibit two-step weight loss during heating process as shows in DTG graph. Weight loss in the DTG graph represents the temperature at which the aluminum alloy machining chips start to melt. The results clearly indicate that TGA curve descends until it becomes horizontal around 850°C. Figure 6 shows that the first, second and third stage rapid weight loss occurs in the temperature range of 23°C–106°C, 106°C–350°C and 350°C–850°C, respectively is assigned to the thermal decomposition of aluminium alloy chips [24].

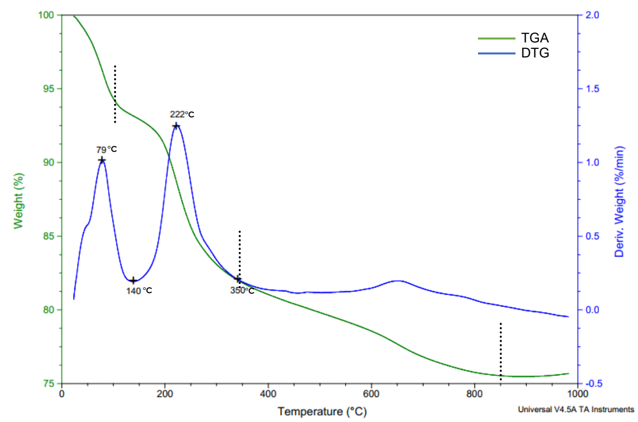


Figure 6 TGA-DTG analysis of aluminium alloy chips from room temperature to 900°C

4.0 CONCLUSION

It is concluded that the (NaCl-KCl) flux salt application at ratio Al:flux (1:0.2) reduced the oxidation during the in-situ casting and promoted the fusion of aluminium alloy chips during an in-situ casting at the heating temperatures of 650°C and 700°C. SEM and EDS analysis proved that the fusion of aluminium alloy chips was hindered by the oxide layer covering the chip's surface due to oxidation of the aluminium alloy chips at elevated temperatures during in-situ casting. XRD analysis confirmed the oxide layer consists of its hydroxides and gamma alumina after being heated at elevated temperatures. Diaspore [a-AlO(OH)] and gamma alumina [γ-Al_2O_3] was presented in the heated aluminium alloy chips sample.

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Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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