



Characteristics of Oxide Layer Formed on the Aluminium (LM6) Alloy and Aluminium (ADC12) Alloy During In-Situ Melting

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Abstract: In-situ melting, also known as an in-situ casting, is a new casting technique that reduces the formation of porosities in aluminium (Al) castings by removing the need for pouring. The melting phenomena of Al are influenced by its oxidation during in-situ melting. The purpose of this research is to find out how the oxide layer generated on the surface of Al alloy (LM6) and (ADC12) cubes during in-situ melting works. Al alloy cubes with approximate dimensions of (L: 1.0cm, W: 1.0cm, and H: 1.0cm) were inserted in a ceramic plate and heated for 30 minutes at 700°C and 750°C in a laboratory furnace. The heated samples were prepared for microstructure and elemental analysis using Optical Microscope (OM), Scanning Electron Microscope (SEM) equipped with Energy Dispersive Spectroscopy (EDS), and X-ray Diffraction (XRD). Both Al alloys deformed from a solid uniform cube to a distorted shape after heating at temperatures of 700°C and 750°C while the colour changed from a shiny and silvery grey to dark grey. Al LM6 alloy cube changed from its shiny and silvery grey (raw sample) to dusty and dark grey mixed with buff (yellow-brown color) at 700°C, while at 750°C it turned to charcoal grey. For Al ADC 12 alloy sample, the colour changed from its shiny and silvery grey to slightly darker grey at 700°C and mixed with yellowish-grey color at 750°C. The colour changes indicating oxidation of the Al alloys cubes during in-situ melting had occurred, forming superficial layers of aluminium oxide Al_2O_3 . XRD analysis revealed the oxide compounds on the surface of Al LM6 alloy cubes were complex oxide including Al_2O_3 hydroxide and spinel. While XRD analysis for Al ADC 12 alloy oxide after heating at the temperature of 750°C contains aluminium oxide hydroxide. It is considered that the aluminium alloy was transformed to the final or stable phase of aluminium oxide (Al_2O_3).

Keywords: Aluminium alloy LM6, Aluminium alloy ADC12, oxidation of aluminium alloy

1. Introduction

Aluminium (Al) alloy is widely used in a variety of sectors, including transportation and automotive, building, electrical cables, home items, and precision engineering components due to its unique properties. The characteristics are low density, lighter than steel, simple to produce, good ductility, strong electrical and thermal conductivities, and corrosion resistance [1]. Al can be manufactured via various manufacturing processes to produce products, parts and components. Casting is a manufacturing technique that is distinct from machining, extrusion, forging, and welding. All

this procedure is constantly used in the production of aluminium alloy goods. However, the casting process is a suitable choice for manufacturing aluminium alloys because it is adaptable in ways that other methods aren't. Investment casting is one of the casting processes used to create Al casts parts with excellent surface quality, near dimensional tolerances, complex contours, and intricate forms [1-2].

The problem of producing quality castings is due to the nature of Al that has a high affinity to oxygen leads to the formation of porosity in the castings decreasing its mechanical performance. A new alternative technique called in-situ melting or in-situ casting is a pouring-free casting process. The technique utilizes a basic idea that permits the metal in smaller form charged in a mould to be heated in a furnace, and the liquid metal later fills the mould cavity as the metal melts [3-4]. After cooling, the solidified casting is removed by opening the metal mould [5] or breaking the ceramic investment casting mould [3-4]. Due to encouraging findings in porosity reduction, in-situ melting, or in-situ casting has the potential to be explored further for investment casting of Al alloys [3, 5]. Therefore, this study aims to explore the characteristics of the superficial oxide layer on Al (LM6) alloy and Al (ADC12) alloy cubes as input for investment casting by in-situ melting.

2. Literature Review

2.1 Aluminium Silicon Casting Alloys

Aluminium (Al) alloys have been increasingly popular in the automobile industry in recent years. This is due to the alloys' properties, which include a high strength-to-weight ratio, good wear resistance, low density, and a low coefficient of thermal expansion. Al-Si alloys were used in a variety of automobile engine components. Silicon (Si) alloys were added to Al as a primary alloying element to lower the thermal expansion coefficient, boost corrosion and wear resistance, and improve casting and machining properties. When the Al-Si alloy solidifies, primary aluminium develops and grows in dendrites. Fig.1 shows an aluminium silicon phase diagram showing the eutectic point of aluminium silicon is at 12.6% silicon and the melting temperature of aluminium is 660°C [5].

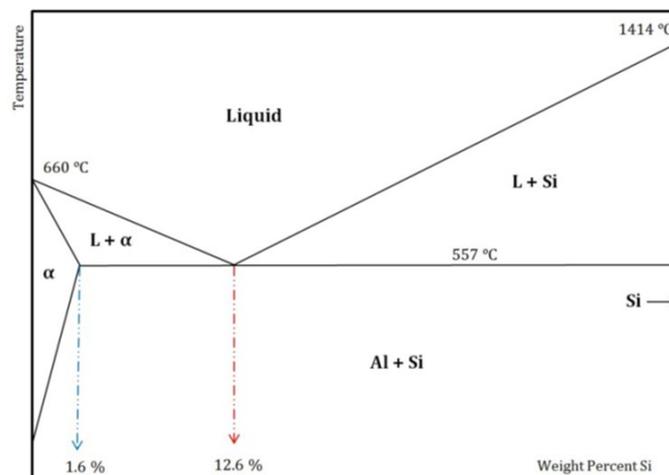


Fig. 1 - The schematic phase diagram of Al-Si [5]

2.2 Physical and Mechanical Properties of Al (LM6) and Al (ADC12) Alloys

Table 1 shows the physical characteristics of Al (LM6) of density, melting temperature, thermal conductivity, specific heat, elastic modulus, coefficient of thermal expansion, and poison ratio [6]. Table 2 shows the physical characteristics of Al (ADC12), including density, melting temperature, thermal conductivity, specific heat, elastic modulus, coefficient of thermal expansion, and poison ratio [7].

Table 1 - Physical properties of Al (LM6) alloy [6]

Physical Properties	LM6 Alloys
Coefficient of Thermal Expansion, (<i>per</i> °C at 20 – 100°C)	20.0 × 10 ⁻⁶
Thermal Conductivity, (<i>Cal/cm²/cm/°C</i> at 25°C)	0.34
Electrical Conductivity, (% <i>copper standard</i> at 20°C)	37
Solidification Shrinkage, (<i>Approx. %</i>)	3.7
Freezing Range, (<i>Approx. °C</i>)	575-565
Pouring Temperature, (<i>Approx. °C</i>)	725

Table 2 - Physical properties of Al (ADC12) alloys [7]

Physical Properties	ADC12 Alloys
Density, (g/cm^3)	2.76
Melting Temperature, ($^{\circ}C$)	660
Thermal Conductivity, (W/mK)	10-40 at 1100 $^{\circ}C$
Specific Heat, (J/gK)	96.2
Elastic Modulus, (GPa)	68.90
Coefficient of Thermal Expansion, (K^{-1})	31.1×10^{-6}
Poisson Ratio	0.33

Many significant attempts are being undertaken to increase the mechanical strength capabilities of Al. Al is alloyed with various alloying elements such as Cu, Fe, Zn, Mg, Si, Mn, and others to increase its mechanical strength and modulus. Al-Si alloys are widely utilized among the numerous aluminium alloys due to their features such as low coefficient of thermal expansion, good bearing capabilities, high corrosion resistance, and sufficient strength, and are therefore commonly employed in aerospace and car structural components. The composition includes the percentage of Aluminium (Al), Copper (Cu), Magnesium (Mg), Iron (Fe), Nickel (Ni), Zinc (Zn), Manganese (Mn) and Silicon (Si). The percentages of the composition are all stated in Tables 3 (LM6) and Table 4 (ADC12) respectively [7,8].

Table 3 - Composition of Al (LM6) alloys [7,8]

Composition of Al (LM6)	Percentage %
Al	Remainder
Cu	0.1
Mg	0.1
Fe	0.6
Ni	0.1
Zn	0.1
Mn	0.5
Si	10.0-13.0
Lead	0.1
Tin	0.05
Titanium	0.2

Table 4 - Composition of Al (ADC12) alloys

Composition of Al (ADC12)	Percentage %
Al	84.79
Cu	2.17
Mg	0.12
Fe	0.73
Ni	0.18
Zn	0.95
Mn	0.13
Si	10.93

Based on phase solubility, many alloys respond to heat treatment. Solution heat treatment, quenching and precipitation, and age hardening are some of the treatments available. Other materials can be work-hardened by mechanical reduction, which is generally combined with other annealing processes for property improvement. These alloys are known as non-heat treatable alloys or work hardening alloys [8]. Tables 5 and Table 6 demonstrate the mechanical characteristics of Al (LM6) and Al (ADC12) alloys, including tensile and yield strength values [7,8].

Table 5 - Mechanical properties of Al (LM6) Alloys [7,8]

Mechanical Properties	LM6 Alloys
Tensile Strength, MPa	73.88

Table 6: Mechanical properties of Al (ADC12) Alloys [7,8]

Mechanical Properties	ADC12 Alloys
Tensile Strength, MPa	310
Yield Strength, MPa	150

2.3 Microstructure of Al-Si (LM6) and Al-Si (ADC12) Alloys

In comparison to many other metal alloys, aluminium alloys have very diverse microstructures. This heterogeneity is caused by alloy additions and impurities combining to form both the intended microstructure and unwanted, large particles known as component particles and residual impurity particles with varying compositions [9]. The morphology observed in the aluminium alloy LM6 is silicon in eutectic alloys. The silicon microstructure in the eutectic might be unaltered (acicular, massive, rod, angular, faceted, flake), partially or modified (fibrous, modified angular), or over modified. If the cooling rate (quench modification) or chemical addition (chemical/impurity modification) are changed, the outcome will be different. It will be developed using the eutectic silicon-crystal process, but the overall effect on the microstructure and the alloy's properties will be the same. Fig. 2 shows aluminium silicon (Al-Si) eutectic alloy microstructure.

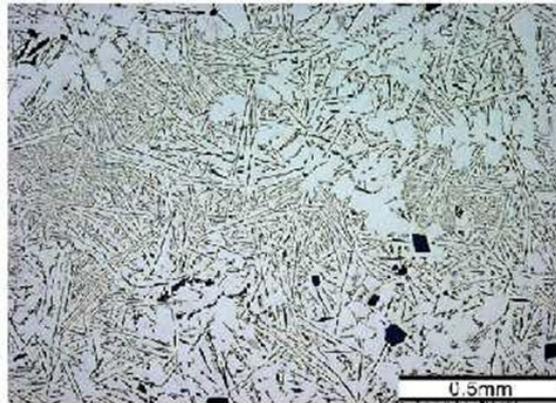


Fig. 2 - Unmodified aluminium silicon eutectic alloy, with primary aluminium dendrites, primary silicon crystals and coarse, acicular eutectic silicon [9]

Aluminium dendrites with dendritic arm spacing in the range of 25 microns are visible in the microstructure of aluminium silicon alloy (ADC12) alloy formed in a cast-iron mould. Fig. 3 shows the solidification of eutectic silicon in the interdendritic area and surrounding the dendrites. Fig. 4 shows a higher magnification micrograph of plate-shaped eutectic silicon and other intermetallic phases. Plate-shaped eutectic silicon is typically 20-25 micron long and 2-5 micron wide. In some cases, eutectic silicon is needle-shaped [7].

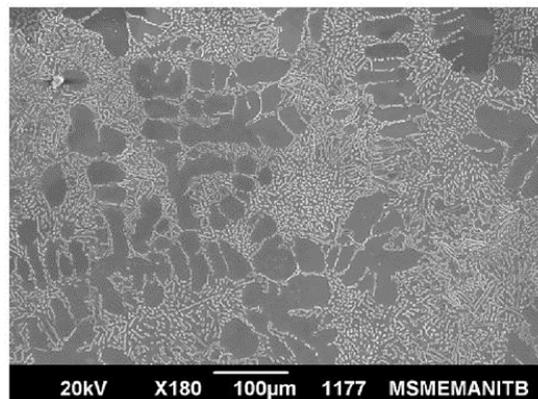


Fig. 3 - SEM micrograph of Al cast (ADC12) alloy showing silicon solidifying in the inter-dendritic region and around the dendrites [7]

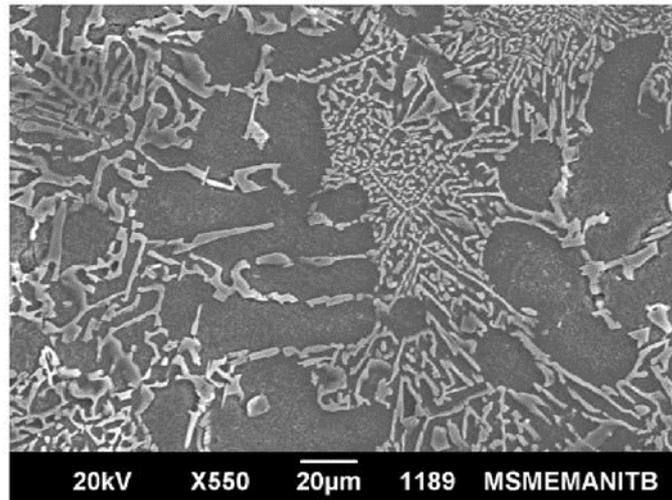


Fig. 4 - SEM micrograph of Al cast (ADC12) alloy showing plate-shaped eutectic silicon and the other intermetallic phases [7]

2.4 Oxidation of Aluminium Alloys at High Temperature

The oxidized aluminium's physical characteristics were defined by its colour change and shape [10]. The oxidized aluminium's appearance altered from metallic-like silver to a matte grey surface. Meanwhile, the colour of the oxides was affected by the heating temperatures. The colour of the heated surface changes from silver-grey to blue-green-yellow-camel-rose-pink-purple-grey above the melting temperature of aluminium, which is 660°C. The colour range agreed well with others, and the colours of the aluminium surface are connected to the phases of the thin superficial aluminium oxide [11].

3. Methodology

3.1 The preparation of Aluminium Silicon Alloy (LM6) and (ADC12) Cubes

Al alloys ingots of (LM6) and (ADC12) were cut to cube sizing approximately (L:1.0cm, W:1.0cm, and H:1.0cm), as shown in Fig. 5. The size of the cubes varied slightly due to the manual cutting operation using a metal saw. The cubes were cleaned from metal burrs and foreign materials before charged into investment casting ceramic plate.

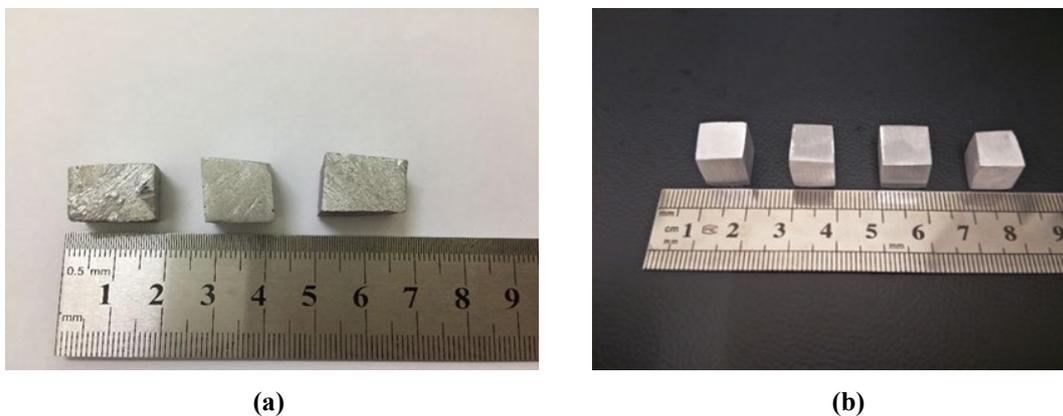


Fig. 5 - Raw samples of (a) Al (LM6) alloy cube; (b) Al (ADC12) alloy cube

3.2 The treatment process of Aluminium Silicon Alloy (LM6) and (ADC12) Cubes

Fig. 6 (a) and Fig.6 (b) show the (LM6) and (ADC12) alloy cubes were placed in an investment casting ceramic plate and loaded in the laboratory furnace for the in-situ melting process. The alloy cubes were heated for 30 minutes at temperatures of 700°C and 750°C as shown in Table 7. The samples were placed at the middle of the furnace to cool to less than 100°C before being removed to cool further at room temperature. The after heated alloy cube samples were then prepared for metallography samples.



Fig. 6 - (a) Al alloy cubes charged into investment casting ceramic plate; (b) loading samples at the middle of the laboratory furnace

Table 7- Heating temperature and time for the experiment

Heating Experiment	Temperature (°C)	Time (min)
1	700	30
2	750	30

3.3 Microstructure and Oxide Analysis

The microstructure of the raw and heated Al (LM6) alloys and (ADC12) alloys cubes were examined using an optical microscope (OM), as shown in Fig.7 (a). Elemental analysis was conducted using a scanning electron microscope (SEM) with Energy Dispersive Spectroscopy (EDS) as shown in Fig.7 (b) to examine the microstructure, oxide structure and morphology, as well as the elemental analysis. The samples were also examined using X-ray Diffraction (XRD) to determine the oxide compound and its phases on the surface of the Al alloys cube samples as shown in Fig.7 (c).

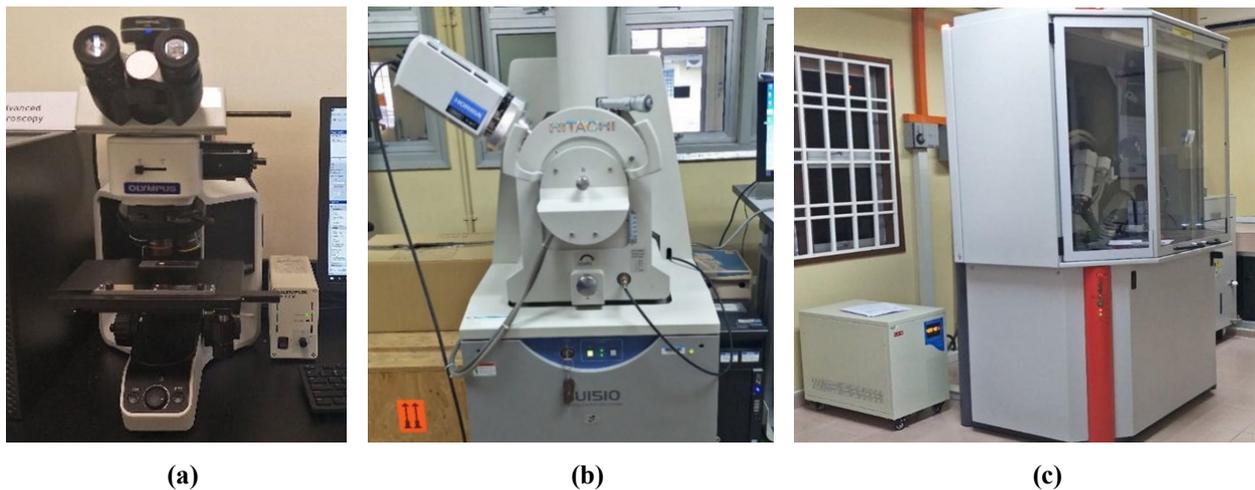


Fig. 7 - (a) Optical Microscope (OM); (b) Scanning electron microscope (SEM); (c) X-ray diffraction (XRD)

4. Results and Discussions

4.1 Physical Properties

The raw or unheated Al LM6 alloy cube had silvery grey physical characteristics and a shiny appearance. However, after 30 minutes of heating at 700°C and 750°C, the cube samples change colour to grey mixed with yellow-brown and then to charcoal grey, as shown in Fig. 8 (a) and (b). The geometry of the LM6 alloy cube deformed from a solid uniform cube to a distorted shape and expanded after being heated at both temperatures. The cube's physical characteristics changed during in-situ melting, indicating that the Al LM6 alloy cube had transformed its phases from solid to semi-solid condition (partially liquid) but oxidized throughout the heating process. The cubes were oxidized proven by its colour

changed from silvery grey and glossy to darker grey and dusty attributed to the oxide crystalline state, which is dependent on the oxide phases [3].



Fig. 8 - Macroscopic images of the Al (LM6) alloys after heated at the temperatures of (a) 700°C; (b) 750°C for 30 min

After 30 minutes of heating at 700°C, the heated Al (ADC12) alloy cube samples changed the shape from a homogeneous cube to a deformed cube, as illustrated in Fig.9 (a). Similar results were obtained when the (ADC12) alloy cubes were heated at 750°C for 30 minutes (Fig. 9 (b)). The deformation of the heated (ADC12) alloy cube indicates that the cube was in-situ melted or at least partially melted due to the cube body expanding during heating. The cube's original shape was preserved despite being distorted, possibly owing to the oxidation of its surfaces, which created an oxide layer that encapsulated the melted section of the cube in the oxide encapsulation, also known as the oxide crust which in agreement with previous research [12].

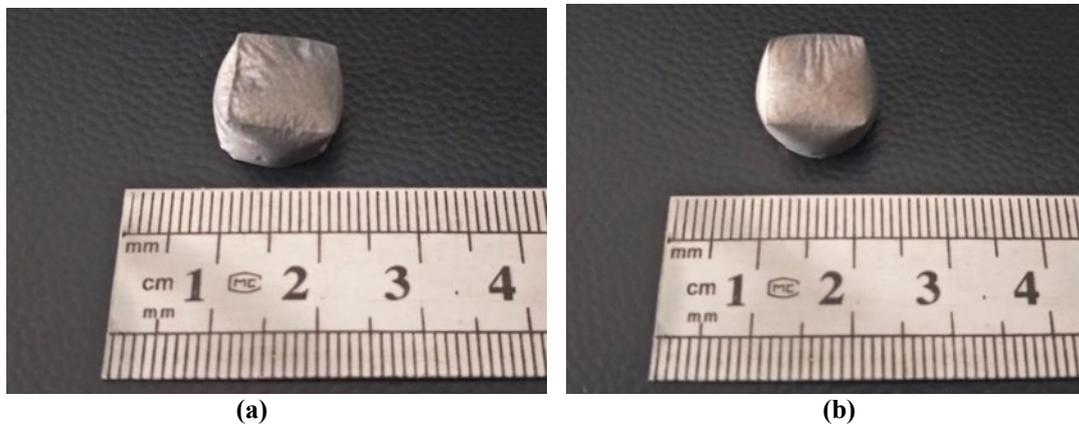


Fig. 9 - Heated ADC 12 cube sample (a) Cube heated at temperature 700°C; (b) Cube heated at temperature 750°C

Fig. 9 (a) shows the colour of the heated (ADC12) alloy cube changed from shiny silvery grey to grey mix with brown after being heated at 700°C for 30 minutes. The colour of the heated samples changed from shiny silvery grey to pearl grey, as seen in Fig.9 (b). The heated samples' appearance was identical for both temperatures of 700°C and 750°C for 30 minutes, with a matte or non-glossy finish. The oxidation that occurred during the cube's heating in the furnace developed a superficial oxide layer on the cube's surface, turning the glossy finish to dusty which is agreed by the previous researcher that the change of the Al alloy colour was a result of the oxide crystalline state depending on its phases [3, 10].

4.2 Microstructure of the heated LM6 and ADC12 Al Alloy Cube

Fig. 10 (a) and (b) show the microstructure of LM6 and ADC 12 Al alloys observed using an optical microscope (OM) at a magnification of 2.5 X 200µm respectively. The result agrees with microstructure of the aluminium alloy (LM6) consisting of α -Al matrix and eutectic silicon flakes [3]. The microstructure of the heated Al LM6 alloy cube sample at 700°C (Fig. 10 (a)) and 750°C (Fig. 11 (a)) was coarser than the raw sample (Fig.10 (a)). A similar observation was found for ADC12 Al alloy cube sample, the microstructure of the heated cubes (Fig. 11(b)) at the temperature of 700°C and (Fig. 12 (b)) at the temperature of 750°C was coarser than the microstructure of the raw sample (Fig.10 (b)) due to the heated samples were slowly cooled to room temperature in the furnace agreed with others [13,14].

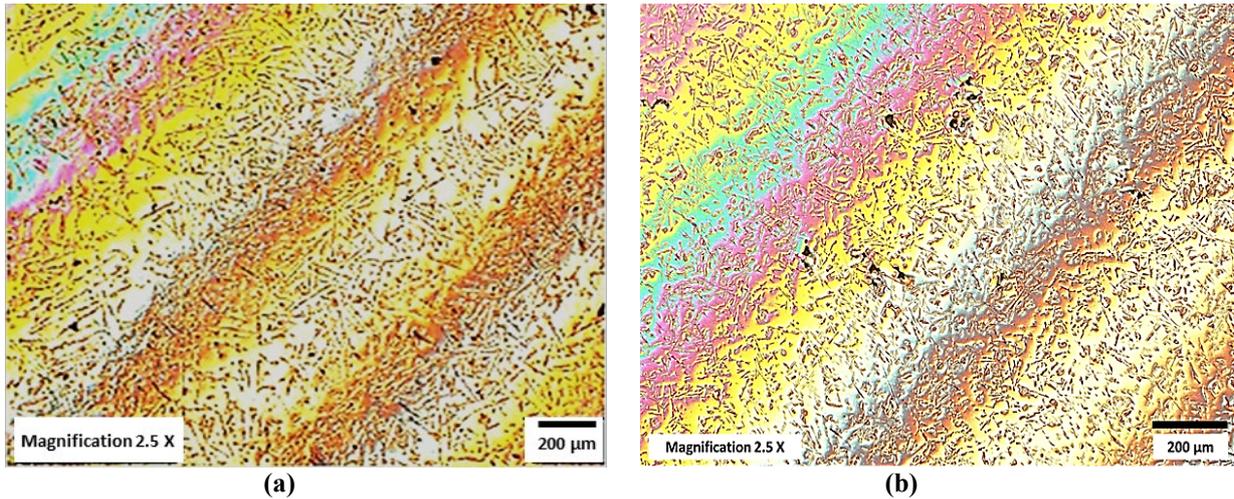


Fig. 10 - The raw alloy cube after polishing (a) LM6 (b) ADC12

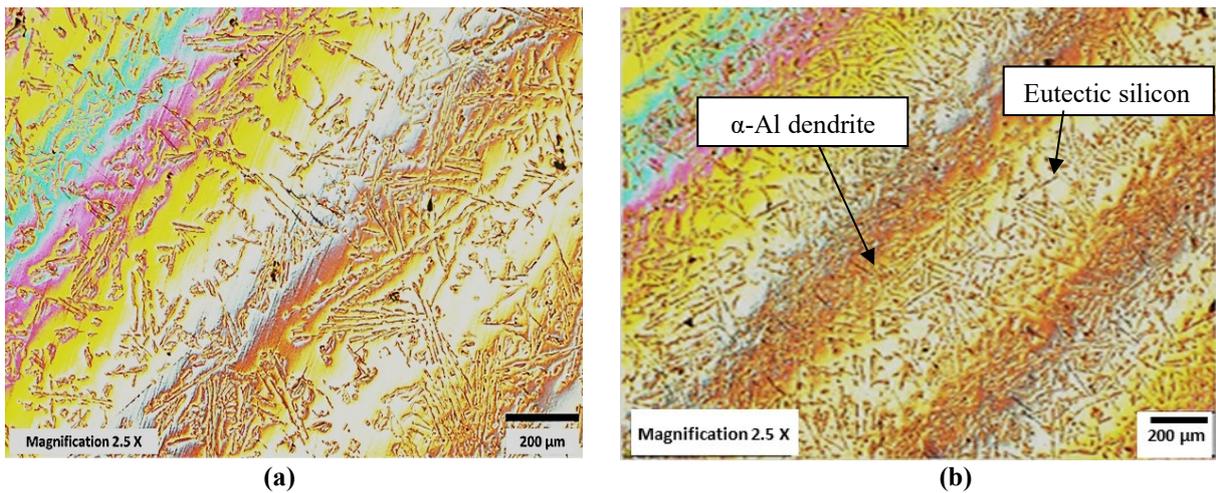


Fig. 11 - The alloy cube heated at temperature 700°C after polishing (a) LM6 (b) ADC12

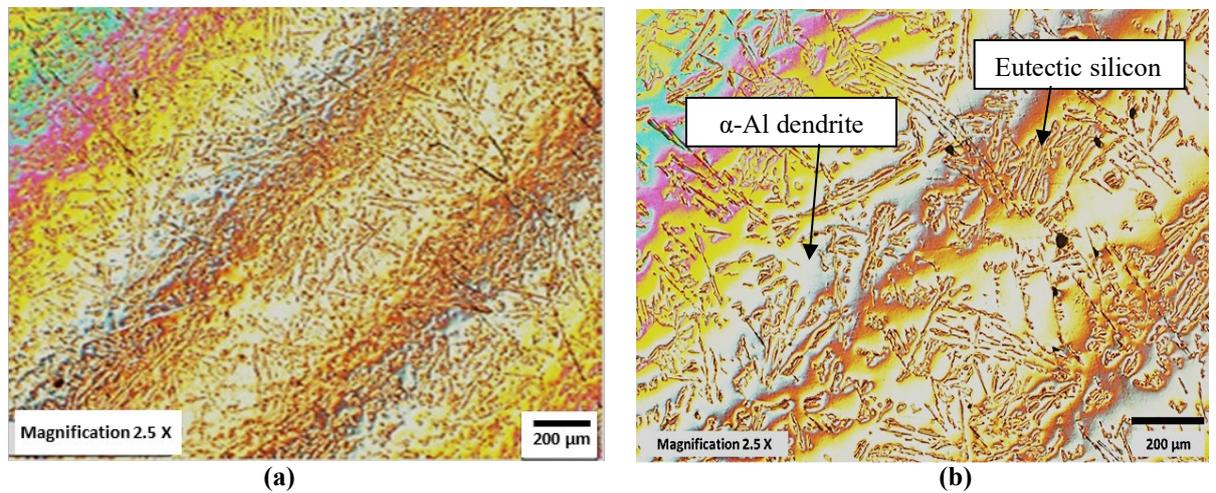


Fig. 12 - The alloy cube heated at temperature 750°C after polishing (a) LM6 (b) ADC12

4.3 Scanning Electron Microscope (SEM) Analysis

Fig. 13 and Fig. 14 show the morphology of a sample heated at the temperature of 700°C and 750°C that portrayed a wrinkled surface. Meanwhile, Fig. 15 (a) EDS spectrum of the heated Al LM6 surface at the temperature of 700°C. Al has the greatest percentage of 60.03wt%, followed by Oxygen (O) at 23.40wt%, Silicon (Si) at 10.67wt%, and Magnesium (Mg) at 5.89wt%. For Al LM6 alloy heated at the temperature of 750°C, Al has the greatest percentage (57.54wt%), followed by Si (21.81wt%), O (17.60wt%), and Mg (3.04wt%) as shown in Fig. 16 (a). The presence of O

on the surface of the heated Al LM6 cube samples indicates the Al cube had oxidized during in-situ melting forming Al_2O_3 agreed with [10].

Fig. 13(b) and Fig.14(b) show SEM image of the oxide morphology that developed on the surface of the ADC12 alloys cube sample after 30 minutes of heating at 700°C and 750°C respectively. The oxide morphology was a wrinkled surface all over the heated cube. Fig.14(b) and Fig. 15(b) show the results of an EDS analysis conducted on the oxide morphology of heated Al ADC12 alloy cube sample using the area selection approach. The spectrum displays peaks of aluminium (Al) and oxygen (O) at 39.79wt% and 25.80wt%, respectively on the cubes sample heated at the temperature of 700°C. While sample heated at the temperature of 750°C shows the aluminium (Al) and oxygen (O) at 46.47wt% and 20.23wt%, respectively. The presence of oxygen on the oxide morphology of the heated Al ADC12 alloy cube sample suggested that the cube had oxidized during heating in the furnace for in-situ melting. However, the complete in-situ melting was hampered due to oxidation of the cube on the surface, which prevented the melted entity in the cube from bursting out and forming a molten pool of alloy. This could be due to the superficial oxide layer on the cube having sufficient strength to withstand the internal tension caused by the molten alloy in it [3, 5, 15].

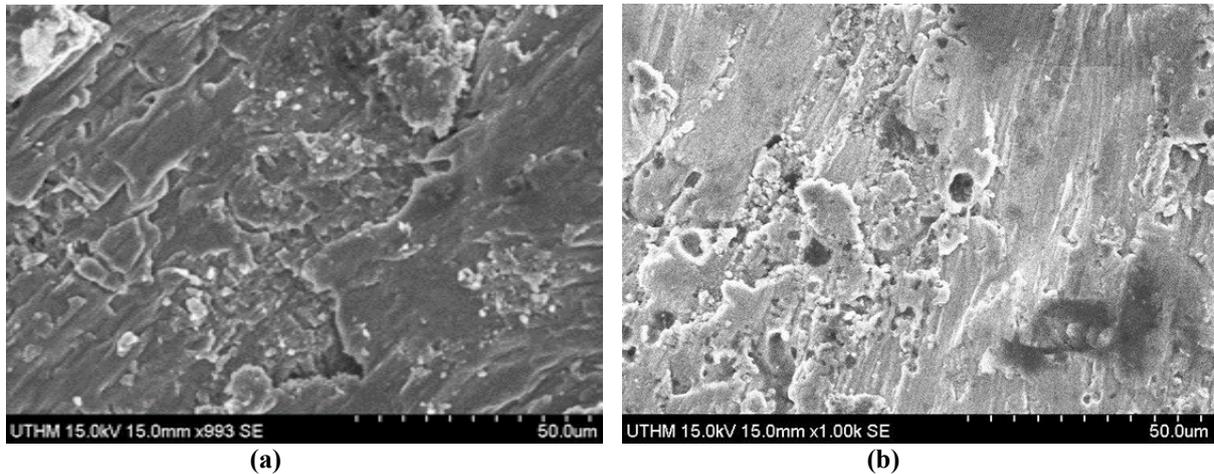


Fig. 13 - Morphology of the alloy cube sample heated at temperature 700°C for 30 min (a) LM6; (b) ADC12

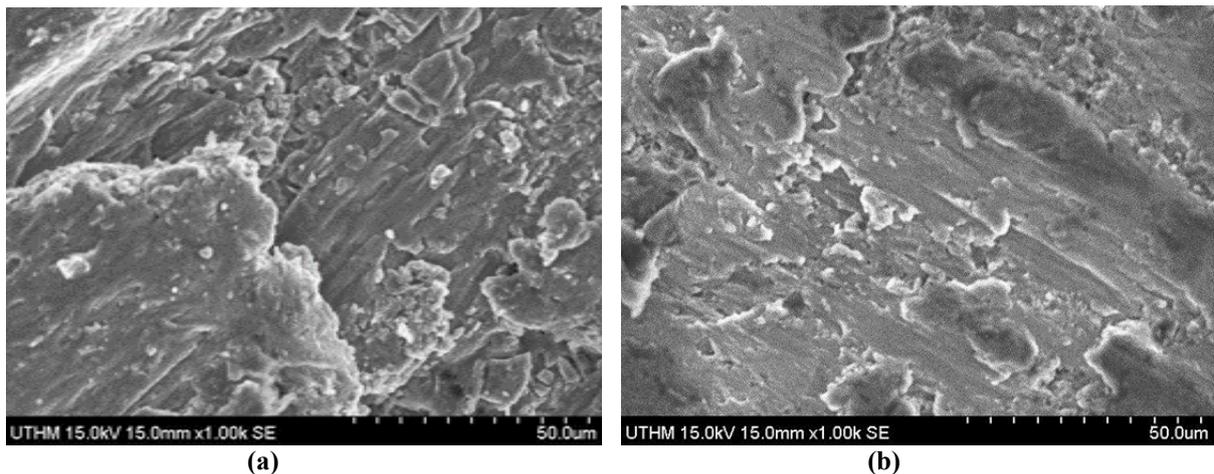


Fig. 14 - Morphology of the alloy cube sample heated at temperature 750°C for 30 min (a) LM6; (b) ADC12

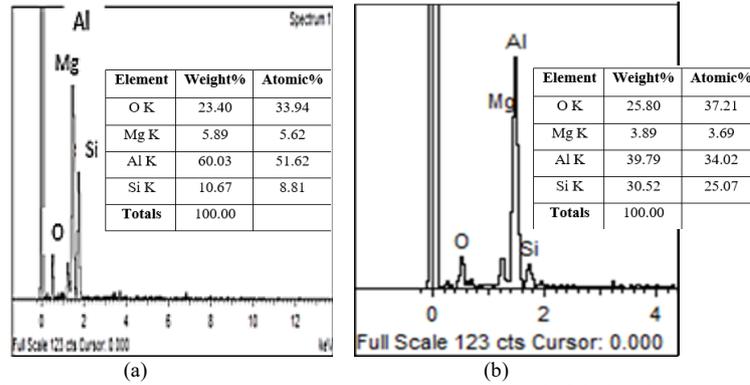


Fig. 15 - EDS analysis of the alloy cube sample heated at temperature 700°C for 30 min (a) LM6; (b) ADC12

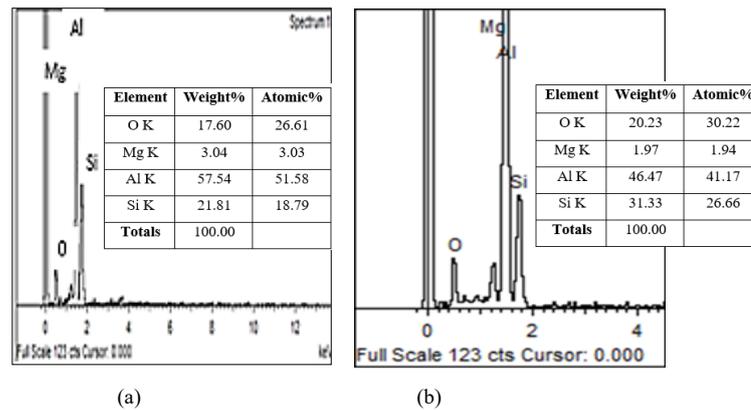


Fig. 16 - EDS analysis of the alloy cube sample heated at temperature 750°C for 30 min (a) LM6; (b) ADC12

4.4 X-ray Diffraction (XRD) Analysis

The XRD spectrum of a raw Al LM6 alloy cube is shown in Fig. 17 revealed five strong peaks of Al at 2θ degrees of 38.47°, 44.72°, 47.30°, 65.10°, 78.23° and 82.46°. The alloying element, Si found at multiple peaks in the spectrum proved that the cube sample was Al-Si. The XRD analysis of the Al LM6 alloy cubes sample heated at the temperature of 700°C for 30 minutes is shown in Fig. 18 demonstrated the presence of spinel (Magnesium Aluminium Oxide) and diaspore (aluminium oxide hydroxide). While the Al peaks was found at 2θ degree of 39.76°, 45.34°, 56.20°, and 79.57°.

Fig.19 depicts the XRD analysis of Al LM6 alloy cubes sample heated at the temperature of 750°C for 30 minutes. Complex oxide compound was discovered based on the XRD spectrum including aluminium oxide hydroxide, diaspore (aluminium oxide hydrate) and spinel (Magnesium Aluminium Oxide). It was found that the alloying element Mg, was also oxidized, forming spinel.

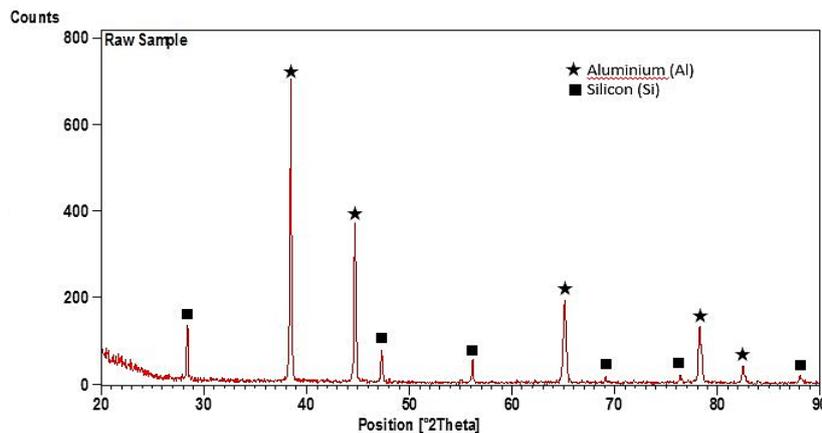


Fig. 17 - XRD analysis of raw or unheated LM6 alloy cube

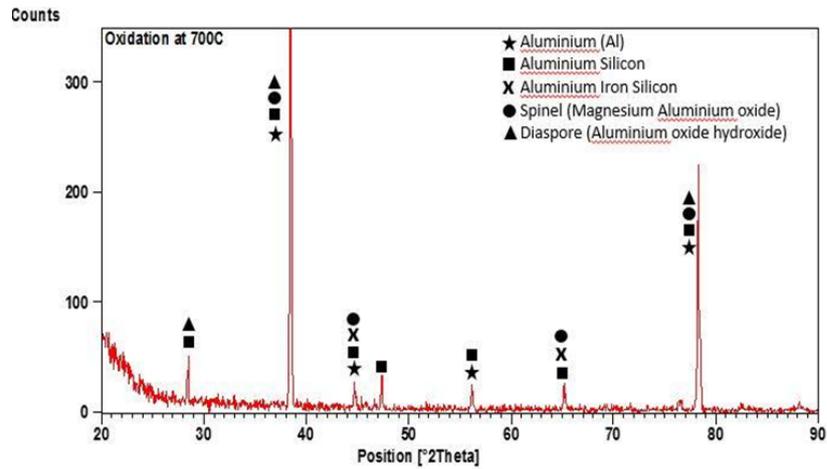


Fig. 18 - XRD analysis of LM6 alloy cube after heated at the temperature of 700°C for 30 mins

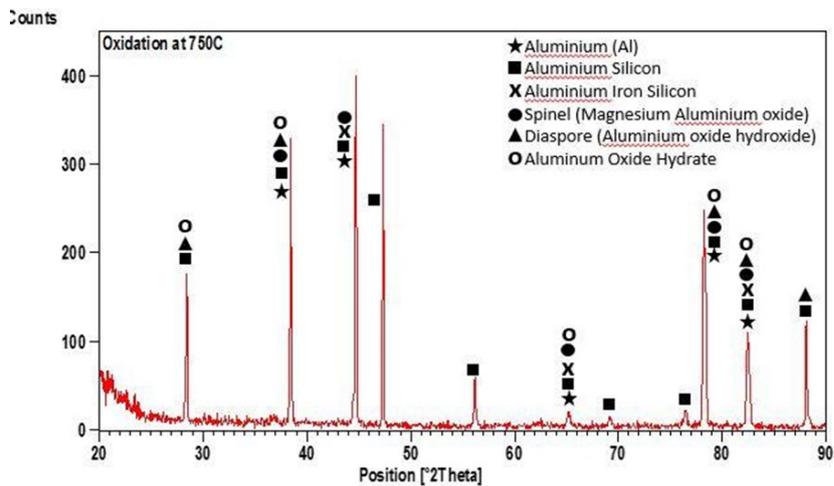


Fig. 19 - XRD analysis of LM6 alloy cube after heated at the temperature of 750°C for 30 mins

The XRD examination of the raw (ADC12) alloy cube before heating is shown in Fig.20. The presence of Al and Si in the sample showed that the alloy was an Al-Si alloy. There were four notable Al peaks at 2θ degrees 38.52°, 44.78°, 65.19° and 78.33°. The Al-Si peaks were also discovered at 2θ degrees 28.48° and 47.38°, whereas the intermetallic complex (Al-Mg-Si) was detected at 2θ degrees 28.48°, 38.52°, 47.38°, and 78.33°. Fig. 20 and Fig. 21 show the XRD analysis of a (ADC12) alloy cube heated for 30 minutes at 700°C and 750°C respectively. There were five strong spinel peaks in Fig. 21 at 2θ degrees 38.52°, 44.70°, 65.20°, 78.40° and 82.50°. The magnesium aluminium oxide hydrate peaks were also found at 2θ degrees 28.48° and 47.50°, whereas the magnesium aluminium silicate peaks were detected at 2θ degrees 28.48°, 38.52°, 56.38° and 82.50°. The diaspore peaks were found at 2θ degrees 28.48°, 38.52°, 56.38° and 82.50°.

Fig. 22 shows four strong spinel peaks at 2θ degrees 38.52°, 44.70°, 65.20° and 78.40°. The magnesium aluminium oxide hydrate peaks were also found at 2θ degrees 28.48°, 47.50°, and 56.3°, whereas the diaspore peaks were detected at 2θ degrees 28.48°, 38.52° and 65.20°. The bayerite peaks were found in positions 2θ degrees 47.50°, 56.38°, 65.20° and 78.40°. Finally, the Aluminium Silicate Hydroxide peaks were found at positions 2θ degrees 28.48°, 47.50°, 56.38°, and 78.40°. The XRD analysis of heated ADC12 cube samples at 700°C and 750°C for 30 minutes revealed no peaks for aluminium oxide (Al_2O_3), the final or stable phase of aluminium oxide. However, significant peaks of aluminium hydroxide, aluminium oxide hydroxide, and aluminium silicate hydroxide were observed in the XRD analysis, as shown in Fig. 21 and Fig. 22, indicating that the Al alloy had oxidized and the Al had been transformed to the stable phase of aluminium oxide. The hydroxide phase identified in the XRD study demonstrates the aluminium-to-aluminium oxide transition process (Al_2O_3). Fig. 21 and Fig. 22 show that the alloying elements oxidized during heating at temperatures of 700°C and 750°C, respectively. Spinel (Magnesium Aluminium Oxide) and its oxide hydrate were identified in the XRD examination of both samples. While the silicon in the alloy oxidized, silicate was formed, as seen in the XRD analysis in Fig. 21 and Fig. 22. These findings revealed that the alloying components in the ADC12 alloy cube samples oxidized during heating in the furnace for in-situ melting application, which is consistent with the literature [13,14].

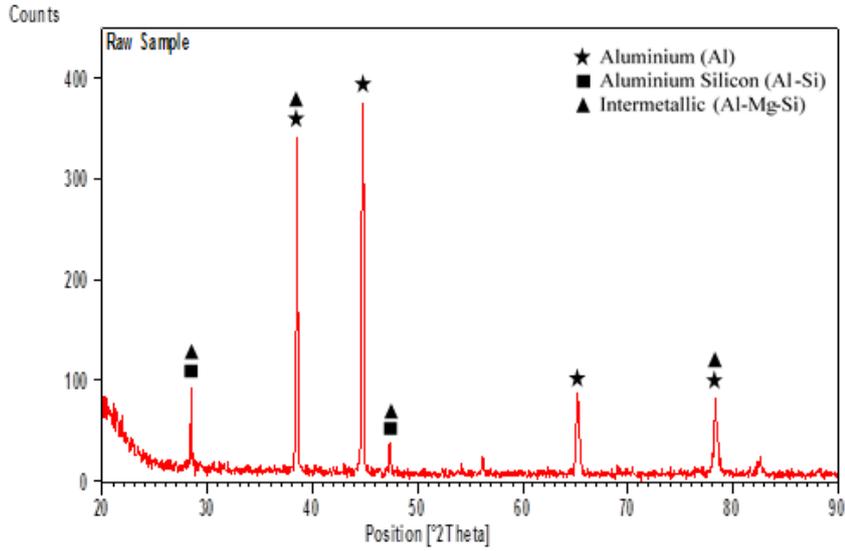


Fig. 20 - XRD analysis of raw (ADC12) alloy cube sample before heating

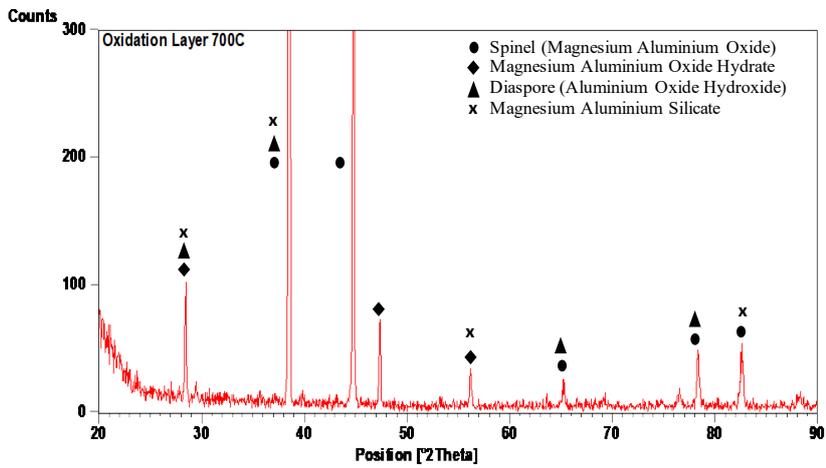


Fig. 21 - XRD analysis of (ADC12) alloy cube sample after heated at the temperature of 700°C for 30 mins

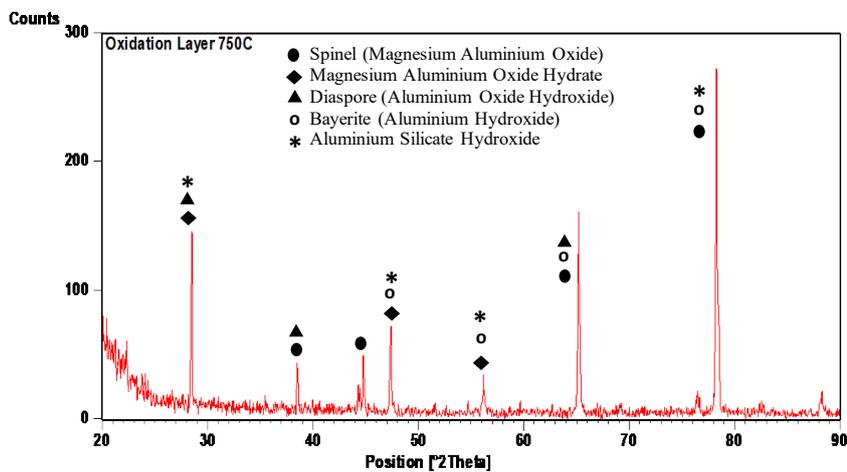


Fig. 22 - XRD analysis of (ADC12) alloy cube sample after heated at the temperature of 750°C for 30 mins

5. Conclusions

In conclusion, the physical characteristics of the heated Al LM6 and ADC12 alloy cube changed from a solid uniform cube to a distorted shape. At the temperature of 700°C, the colour of the heated cubes changed from silvery grey to a grey mix with golden brown, and then to charcoal grey at the temperature of 750°C. While the ADC 12 alloy cubes changed from shiny silvery grey to slightly darker (700°C) and to pearl grey after heated at the temperature of 750°C. The oxide developed on the surface of the Al LM6 and ADC12 alloy cubes portrayed wrinkle appearance, where Al and O elements were discovered by SEM and EDS analysis, indicating the morphology was Al₂O₃. XRD examination revealed that the oxide compounds that formed on the surface of the Al LM6 alloy cubes were complex oxides including Al₂O₃ hydroxide and spinel. The temperature of 750°C resulted in the formation of more complicated oxide compounds. The alloying elements, magnesium and silicon, were also oxidised, resulting in magnesium aluminium oxide and silicate. The SEM examination revealed that the Al ADC12 alloy was oxidised, developing Al₂O₃, also the XRD analysis revealed the existence of aluminium oxide hydroxide, suggesting that the Al alloy was transformed to the final or stable phase of aluminium oxide (Al₂O₃).

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