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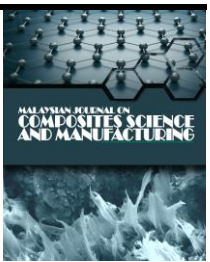

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T6 Heat Treatment Optimization of Thixoformed LM4 Aluminium Alloy using Response Surface Methodology

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ABSTRACT

Aluminium alloy of LM4 is abundantly utilized-in automotive and aerospace industries because of its good fluidity, high mechanical strength, good wear resistance and low density. T6 heat treatment as part of its forming process will enhanced further these properties. However, the total time required for T6 is very long and often delay the overall production cycle time. Furthermore, thixoforming of semi-solid processing method, which promises a near-net-shape and further enhanced performance of the end products. One of the major concerns is an additional cost in billet feedstock production that slows down the application in industries. Therefore, this paper aim is to optimize the T6 heat treatment on the thixoformed LM4 alloy using response surface methodology (RSM) of Design Expert software. Thus, a simple cooling slope casting to produce the feedstock for thixoforming of near globular and fine α-Al microstructure is being applied.Cooling slope parameters at pouring temperature of 660 °C; slope length of 440 mm and tilt angle of 45 °. As results, small globular grain size of 35.709 µ and shape factor of 0.89 are obtained. Moreover, based on the RSM responses, the optimum parameters of T6 heat treatment; solution treatment at 530 °C for 30 minutes and 2 hours of ageing at 180 °C. The overall strength of LM4 improved significantly with ultimate tensile strength (UTS) of 252.3 MPa, 98.9 HV surface hardness and coefficient of friction (CoF) of 0.4299, respectively.

Keywords:
Semisolid metal processing, Cooling slope casting; Aluminium alloy; T6 heat treatment

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1. Introduction

Al-Si-Cu alloys such as LM4 emerged in the limelight of abundant automotive industries because of the superior mechanical properties. This makes these alloys suitable for components such as cylinder blocks, piston and camshaft cap as stated by Rahman *et al.* [1]. Forming of Al alloy products using conventional casting will always encounter the common problems such as macrosegregation and porosity due to the coarse primary α-Al and non-circular shaped eutectic Si phase existed in the microstructure of Al alloys. Thixoforming relies on methods to obtain non-dendritic microstructure to exhibit thixotropic properties and this statement is reported by previous researchers [1-5].

Kumar *et al*. [6] mentioned that the precondition of semi-solid metal processing is to obtain fine nearly globular primary α-Al particles slurry and this precondition explains on the objective of billet feedstock production by different routes such as mechanical stirring, magneto-hydrodynamic stirring (MHD) and strain-induced melt activated (SIMA) process, as Atkinson [7] listed in her paper. Few authors suggest that the cooling slope is a very simple process to get the primary α-Al phases to transform to desired fine non-dendritic microstructure by pouring superheated melt along a cooling slope plane and immediate cooling in a mould making use of high nucleation rate associating itself with low-temperature casting and chill cooling [8-9]. Optimum parameters of cooling slope casting include the tilt angle, pouring temperature, pouring length and wall temperature. These parameters are crucial to obtain a degree of sphericity or shape factor of primary α-Al closest to 1 and a very fine homogenous globular microstructure as discussed in several available literatures [6, 10, 11].

Heat treatment has proven to have the ability to improve the properties of aluminium alloys in which T6 heat treatment and even modifying T6 heat treatment can increase the strength as well as the hardness of aluminium alloy as proven in both Hanizam's and Fabrizi's research paper [3, 12]. The maximum strength of aluminium alloys are produced from T6 heat treatment but unfortunately, Moller *et al.* [13] stated that the cost and production time is considerably remarkable increased. Fabrizi also mentioned that T6 heat treatment consists of three steps which are solution heat treatment, quenching and age hardening [13]. According to the American Society for Testing and Materials (ASTM B917), the duration of T6 is 9 hours (solution treatment between 6 to 12 hours and ageing between 3 to 5 hours) [14-15].

Even though T6 heat treatment has been popular in aluminium alloys treatment, the optimized parameters of T6 for thixoformed LM4 Al alloy is still unclear. Therefore, this paper presents an original work of investigating the effect of optimizing T6 heat treatment of thixoformed LM4 Al alloy billet feedstock produced by Cooling Slope (CS) method on mechanical properties and wear resistance of the alloy. Pouring temperature, one of the parameters of CS casting was experimented at three different temperatures to generate the optimum thixoforming feedstock before the study on obtaining optimum T6 heat treatment using DOE-RSM method.

2. Methodology

2.1 Differential Scanning Calorimetry (DSC)

Chemical composition of LM4 Al alloy used is as depicted in Table 1. As-received LM4 Al alloy was cut into 2 mm x 2 mm x 1 mm sample size for DSC testing. The sample and reference were tested and thus the heat flow and liquid profile of the alloy was defined. LM4 Al Alloy has a liquidus temperature of 647 °C and a solidus temperature of 536 °C referred to the DSC curve in Figure 1. Therefore, as thixoforming process is generally carried out at a thixotropic temperature of the alloy in which the alloy is at 50% liquid content, the temperature can be estimated from the liquid fraction profile.

Table 1
Chemical Compositions of LM4 Al Alloy by weight percentage (wt.%)

Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	Al
5.31	0.27	3.11	0.01	0.13	0.01	0.01	0.02	0.06	BAL

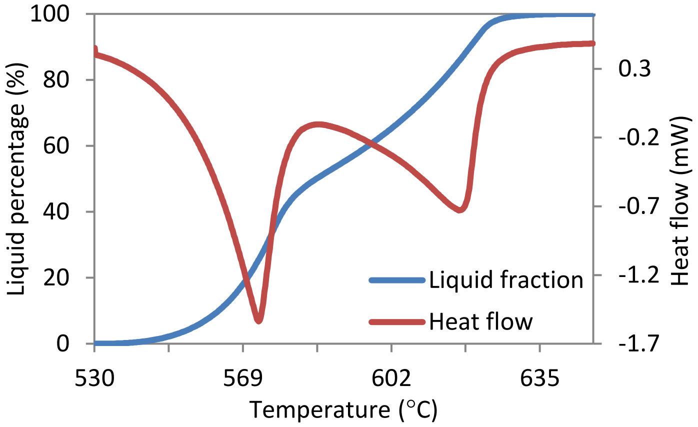


Fig. 1. DSC curve of LM4 Al Alloy

2.2 Cooling Slope (CS) Casting

CS casting parameters of LM4 Al alloy is listed in Table 2. The pouring temperatures of 620 °C, 640 °C and 660 °C was selected around the liquidus temperature (647 °C) which is based on DSC test. The tilt angle and slope length were kept constant at 45 ° and 400 mm respectively as referred to the optimized value [14].

Table 2
CS casting parameters of LM4 Al Alloy

No. of experiment	Pouring temperature, T_p (°C)	Slope length (mm)	Tilt angle (°)	Superheat temperature, T_s (°C)
1	620	400	45	680
2	640	400	45	680
3	660	400	45	680

The alloy was fully melted at a superheat temperature of 680 °C before allowing it to cool down to their specific pouring temperature. As the melted alloy reached its respective pouring temperature, it was immediately poured on an inclined CS stainless steel plane before it was collected at the end of the plane in a mould. At this point, characterization which will be later explained were done on the samples to obtain the best pouring temperature for CS casting before the experiment was continued with the next process.

2.3 Thixoforming Process

The CS casted billet feedstocks was cut to a dimension of 25 mm Ø x 110 mm and placed in the induction coil of T30-80kHz thixoforming machine as shown in Figure 2. Referred to the liquid profiling from the DSC testing, 580 °C will be the perfect thixotropic temperature of the alloy as it corresponds to 50% of liquid content thus, the reheated billet feedstocks were in a semisolid state before being simultaneously rammed into the mould, forming a thixoformed LM4 Al alloy. The reheating process temperature increment was controlled by increasing 50 Hz of current frequency every minute until the desired thixotropic temperature was reached. The billet temperature was measured using a thermocouple. The thixoformed alloys were left to cool down at ambient temperature.

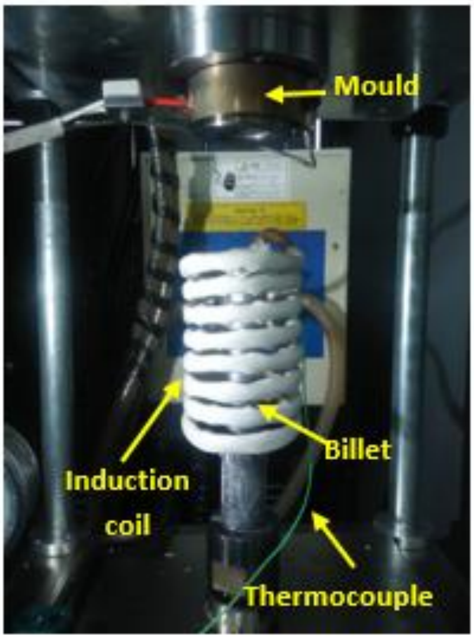


Fig. 2. Thixoforming setup

2.4 T6 Heat Treatment

As the study aims to acquire the optimal T6 heat treatment parameters for thixoformed LM4 Al alloy, RSM method and Design Expert software was used to generate the correlation between the input process parameters and output process responses.

Table 3 shows T6 heat treatment on thixoformed LM4 Al alloy with a total of 29 experiment runs conducted in random. Each experiment run was replicated three times to obtain a reliable result. In addition, five centre points were added, providing stability and possible curvature. A confirmation experiment was conducted based on the optimum results secured from DOE.

2.5 Material Characterisation

Characterizations of the samples are executed using optical microscopy (OM), and scanning electron microscopy (SEM) at several points of this experiment, which are after CS casting, after thixoforming and after T6 heat treatment process.

The samples were sectioned and prepared for OM by grinding using 240, 320, 400, 600, 800 and 1200 grits, polishing with 1 µm and 3 µm diamond solutions, and etching with Keller’s solution respectively.

The OM images of CS casting samples were used to identify and evaluate the grain size and sphericity of α-Al so that the CS casting parameters that could provide small grain size with favourable sphericity could be selected to produce billet feedstock for thixoforming process.

Table 3
Experiment runs of T6 heat treatment optimization

Run	Factor 1	Factor 2	Factor 3	Factor 4
	A: Solution treatment (ST) Temperature (°C)	B: Solution treatment (ST) Time (minute)	C: Ageing Temperature (°C)	D: Ageing Time (hour)
1	530	60	170	4
2	520	60	170	4
3	510	30	180	6
4	510	120	180	2
5	520	60	170	4
6	530	30	180	2
7	510	30	160	2
8	510	60	170	4
9	520	60	170	4
10	520	120	170	4
11	520	60	180	4
12	520	60	170	4
13	510	30	160	6
14	530	30	160	2
15	520	60	170	4
16	510	120	160	2
17	510	120	180	6
18	530	120	160	2
19	530	30	160	6
20	530	120	180	2
21	520	60	170	2
22	530	30	180	6
23	530	120	160	6
24	520	60	160	4
25	530	120	180	6
26	510	30	180	2
27	510	120	160	6
28	520	60	170	4
29	520	60	170	6

2.6 Mechanical Testing

Tensile test was conducted in accordance to ASTM B557M. The samples were machined using an electrical discharge machine (EDM) wire cut as depicted in Figure 3 and Table 4. Hardness test was conducted on Vickers hardness tester (Matsuzawa machine) with a load and dwell time of 10 N and 10 s, respectively.

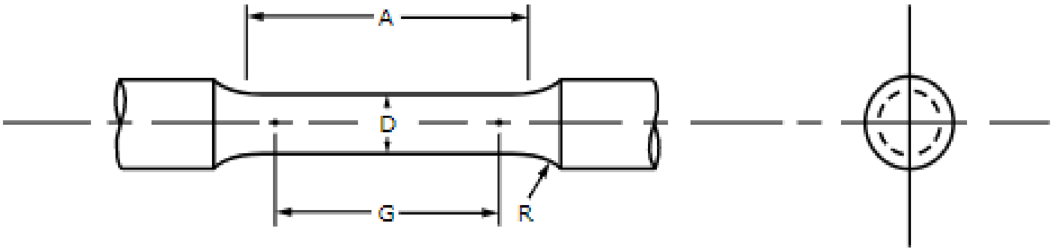


Fig. 3. Small-size Specimen Proportional to the Standard Specimen

Table 4
LM4 Al Alloy Tensile Test Specimen

Section	Dimension (mm)
Nominal diameter	4
G-gage length	20.00 ± 0.04
D-diameter	4.00 ± 0.05
R-radius of fillet	4
A-length of reduced section	24

2.7 Wear Testing

The wear test was conducted according to ASTM G99 based on Table 5, which the samples was cut to a 3 mm Ø x 30 mm. The alloy was mounted on the disc holder and acted as a pin, while SKD II stainless steel used as a counter body.

Table 5
Wear Test Parameters for LM4 Al Alloy

Parameter	Value		
Counter disc type	SKD II		
Normal force (N)	10		
Sliding distance (m)	1000		
Pin-end diameter (mm)	3		
Sliding speed (m/s)	0.1		
Track diameter (mm)	25	30	35
Sliding speed (RPM)	76	64	55
Sliding time (hour:min:sec)	2:47:32	2:45:48	2:45:24

The frictional force data were recorded using WINDUCOM software. The Coefficient of friction (CoF) was calculated using the equation as shown in Eq. (1).

$$\mu = \frac{F}{W} \tag{1}$$

where *F* is frictional force (N) and *W* is applied load (N).

3. Results

3.1 Microstructural Analysis

Fine α-Al dendrite microstructure surrounded by the Si eutectic phase was exhibited by as-received LM4 AL alloy in Figure 4 while Figure 5 shows the comparison of the LM4 Al alloy's microstructure after CS casted at different pouring temperature of 620 °C, 640 °C and 660 °C respectively. Thus, based on the as-received alloy image and the CS casted alloys images, it can be seen that the purpose of CS casting to enhance break off of primary dendritic arms and minor breaking of secondary dendritic arms are accomplished, leading to a spheroid microstructure of billet feedstock. Safian *et al.* performed cooling slope at CS length and tilt angle of 400 mm and 45 ° respectively and concluded that both selected parameters contribute to increment of α-Al nucleation and homogenously distributed nucleus due to uniform cooling [16]. Secondary dendritic arm breaking increased which is observed after thixoforming process.

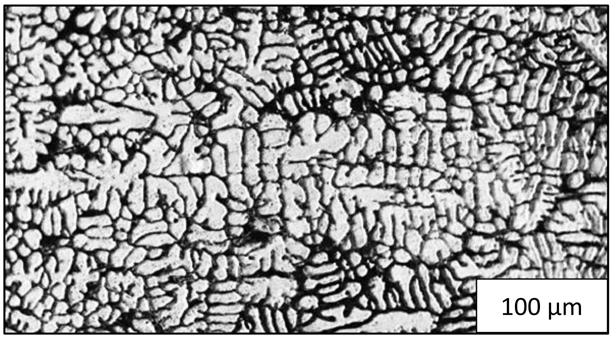
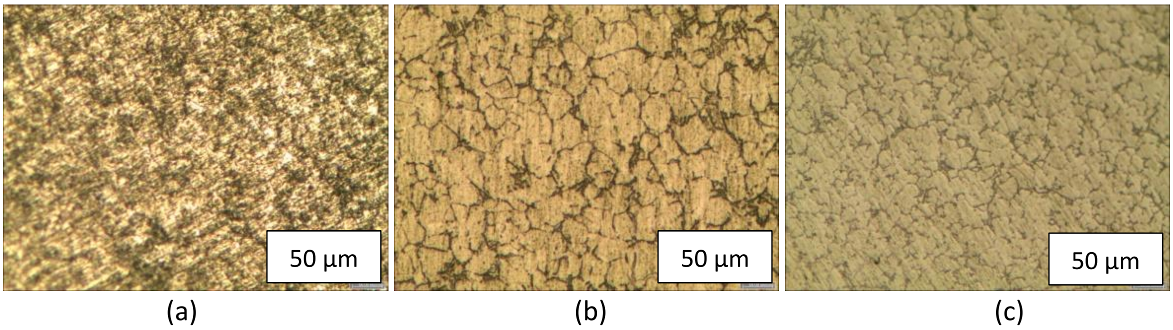


Fig. 4. As-received LM4 Al alloy



(a) (b) (c)

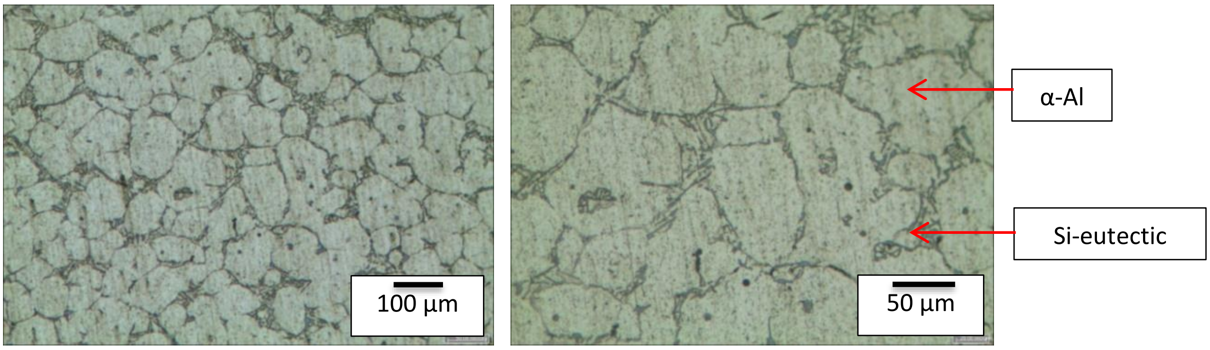
Fig. 5. Microstructure of CS casted LM4 alloy at pouring temperature of (a) 620 °C, (b) 640 °C and (c) 660 °C

As the melted alloy is poured at different pouring temperature, each CS casted sample exhibit different grain size and shape factor. Small grain size and shape factor near to 1 are principal factors to produce ideal billet feedstock as stated by Legoretta *et al.* [17]. According to grain size and shape factor of the casted samples plotted at Table 6, ideal billet feedstock was casted at pouring temperature of 660 °C with a small grain size of 35.709 µm and shape factor of 0.89, which is very close to 1. From the Table 6, the 2nd experiment shows that the lowest average grain size of 27.248 µm at 640 °C. Low pouring temperature produce finer spheroidal structure as reviewed by Kumar *et al.* [18]. The relative shape factor of 1.042 conveys that the sample had low elasticity.

Table 6
Grain size and shape factor (SF) of CS casted alloy at different pouring temperature

Number of experiment	Cooling slope casting parameter			Average grain size (µm)	Shape factor
	Cooling slope temperature (°C)	Tilt angle (°)	Cooling slope length (mm)		
1	620	45	400	86.071	2.594
2	640	45	400	27.248	1.042
3	660	45	400	35.709	0.89

Figure 5 shows the optical microstructure images of as-thixoformed CS casted LM4 AL alloys samples magnified at 50 and 100 times Magnification. The images depicted fine homogenously distributed globular α-Al surrounded by Si eutectic matrix after thixoforming process. Meanwhile, the microstructure evolved due to the shearing force of the CS casted sample during reheating and forming process. A near-spheroidal α-Al phase appeared in Figure 6 through the thixoforming process show that this process is better than connventional forming processes.



(a) (b)

Fig. 6. Optical Microstructure of as-Thixoformed LM4 Aluminium Alloy at (a) 100x Magnification (b) 50x Magnification

Eutectic Si characteristics of Al-Si casting alloys highly influence its mechanical properties. The backscatter electron image in Figure 7 and EDX of thixoformed LM4 alloy micrograph in Fig. 8 displays and confirms the presence of three types of phases formed in the alloy; $Al_5Cu_2Mg_8Si_5$ and Al_2Cu and β-Al_5FeSi. Salleh *et al.* [19] concluded from their study that the iron-rich intermetallic phase (β-Al_5FeSi) appears in needle shape phase and is accountable for reducing the alloys' mechanical properties.

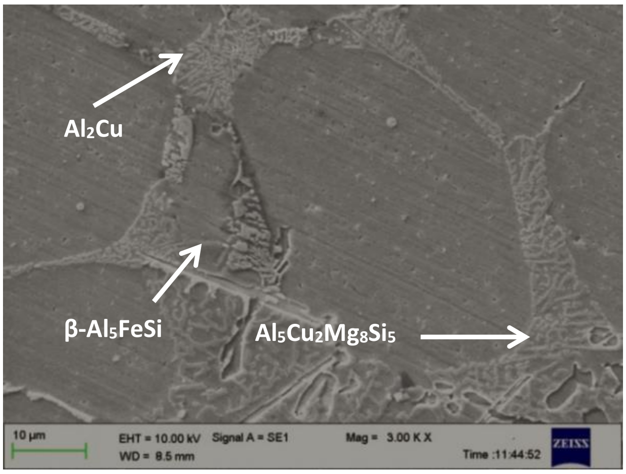
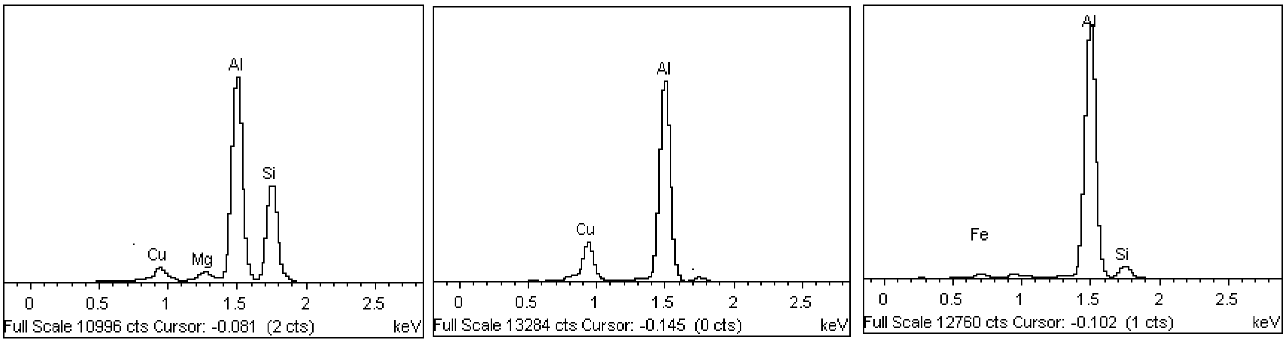


Fig. 7. Backscatter electron image of as-thixoformed LM4 Al alloy



(a) (b) (c)

Fig. 8. EDX of thixoformed LM4 Al alloy micrograph confirms presence of (a)$Al_5Cu_2Mg_8Si_5$, (b) Al_2Cu, and (c) β-Al_5FeSi

3.2 Mechanical Properties

Table 7 shows the results of tensile strength and hardness as response 1 and response 2, respectively. The tabulated response data concluded that Run 6 exhibits the best mechanical properties with tensile strength of 252.7 MPa and hardness of 99.3 HV. The optimum parameters of T6 heat treatment of solution treatment (ST) and ageing temperatures are at 530 °C and 180 °C, and at the lowest times required. This finding is undeniably advantageous for both industries and researchers to reduce the production cost.

Table 7
Experimental Results for Tensile Strength and Hardness

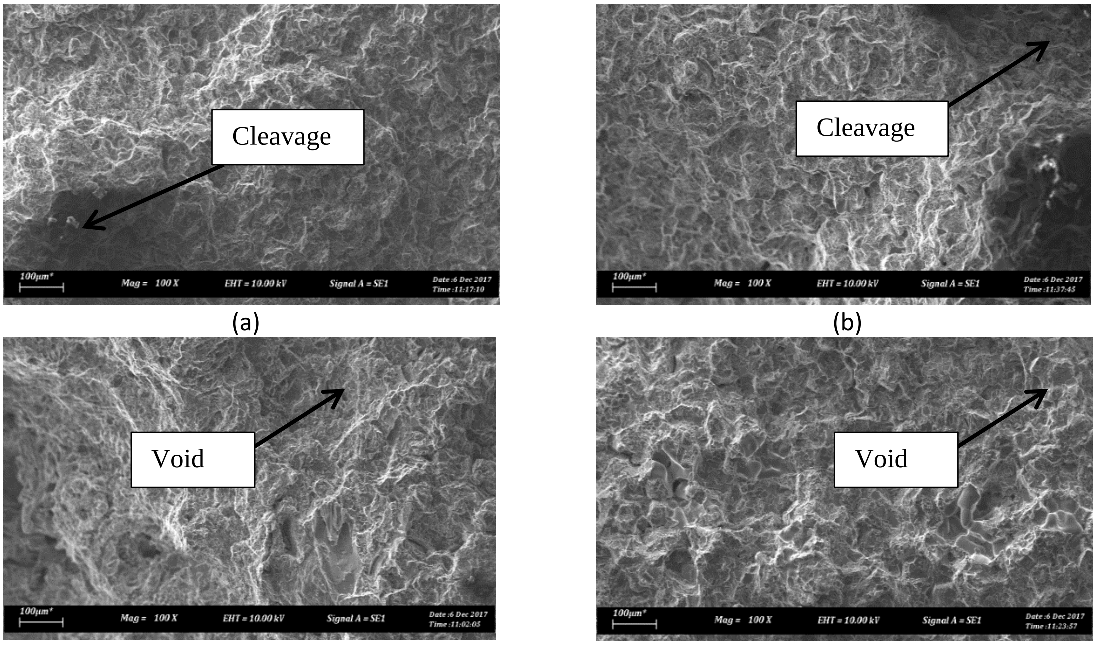
Run	ST temperature (°C)	ST time (minute)	Ageing temperature (°C)	Ageing time (Hour)	Response 1- Tensile Strength (MPa)	Response 2- Hardness (HV)
1	530	60	170	4	236.659	94.2
2	520	60	170	4	239.025	92.7
3	510	30	180	6	234.877	97.4
4	510	120	180	2	220.676	94.4
5	520	60	170	4	235.518	95.1
6	530	30	180	2	252.697	99.3
7	510	30	160	2	212.719	91.1
8	510	60	170	4	232.666	98.4
9	520	60	170	4	235.104	92.6
10	520	120	170	4	238.795	96.2
11	520	60	180	4	238.035	97.4
12	520	60	170	4	235.921	92.9
13	510	30	160	6	231.847	95.2
14	530	30	160	2	237.292	90.3
15	520	60	170	4	237.001	93.2
16	510	120	160	2	212.159	90.2
17	510	120	180	6	249.941	98.2
18	530	120	160	2	238.323	95.9
19	530	30	160	6	233.028	91.6
20	530	120	180	2	247.879	99.1
21	520	60	170	2	237.244	98.3
22	530	30	180	6	241.063	91.7
23	530	120	160	6	242.843	96.7
24	520	60	160	4	233.331	92.6
25	530	120	180	6	251.954	98.6
26	510	30	180	2	227.509	97.7
27	510	120	160	6	237.163	97.9
28	520	60	170	4	233.758	94.8
29	520	60	170	6	240.973	98.5

Table 8 conveys a significant improvement of tensile properties from as-cast alloys to CS casted, thixoformed, T6 thixoformed (ASM standard) and T6-thixoformed (ST-30 min) alloys with 129.7 MPa, 158.3 MPa, 184.5 MPa, 247.9 MPa and 252.3 MPa respectively. Optimization of T6 heat treatment exhibit 36.75% UTS increment from as-thixoformed sample with much shorter time compared to only 34.36% UTS increment for standard T6 heat treatment process.

Table 8
Strength Properties of LM4 Al Alloy

Sample	UTS (MPa)	YS (MPa)	Elongation to fracture (%)
As-cast	129.7	86.8	0.7
Cooling slope	158.3	108.3	1.1
Thixoformed sample	184.5	125.1	2.4
Thixoformed + T6 (ASM standard)	247.9	171.7	2.8
Thixoformed + T6 (ST- 530 °C for 30 min and Ageing-180 °C for 2 h)	252.3	201.8	3.2

Figure 9 represents SEM observations on fracture surfaces of LM4 alloys after the samples undergone tensile force. Due to coarser and rod-like elongated Si particles in as-cast and CS casted samples, the tendency to of recurring fractures are higher as compared to the harder-to-break small round silicon particles in thixoformed samples. This is the evidence of the enhancement made from thixoforming process. From Figure 9 (d), it shows that the cleavage planes mostly disappear and was covered by dimples because of spheroidization of eutectic Si. Thus, T6 heat treated LM4 Al alloys have a higher elongation the fracture properties, leading increment in tensile strength.



(a) (b)

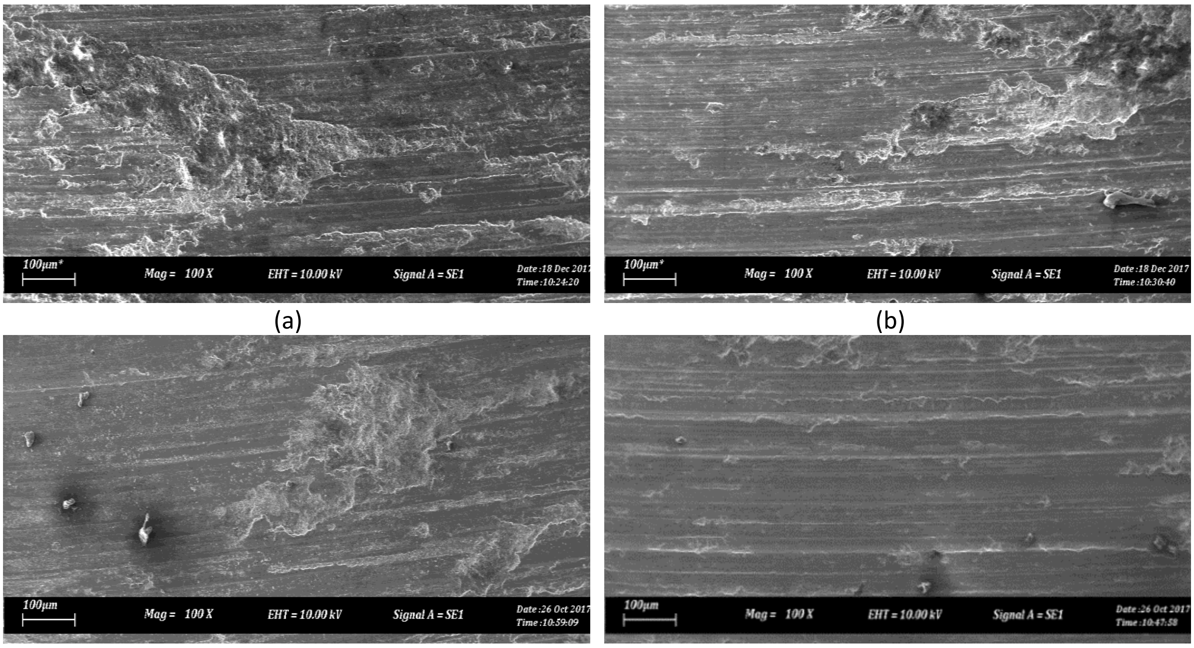
(c) (d)

Fig.9. SEM of tensile test specimens of LM4 Al Alloy (a) As-cast, (b) CS casted, (c) Thixoformed, and (d) T6-thixoformed sample

Surface hardness test, shows that 98.9 HV has the highest reading, which is presented by the optimized T6 heat treated sample with ST of 30 minutes at 530 °C and ageing treatment of 2 hours at 180 °C. The ageing process was responsible at the increasing Si eutectic and α-Al alloys at 180°C precipitation. Longer ST and ageing time increase the surface hardness. As the ageing time increases, the spheroidal microstructure homogenously formed, which is the dislocation hinder between intermetallic and the primary alloy atoms. Higher precipitations in the intermetallic phase construct a higher surface hardness of the alloy. In another study, Hanizam *et al.* [20] also mentioned that the transformation of eutectic-Si particles play critical role in enhancing the mechanical properties of a metal composite. In this study, smaller grain size obtained through the optimum CS casting contributes to the increasing hardness compared to conventional casting.

3.3 Wear

Figure 10 shows the abrasive wear mechanism for all samples. The images exhibit that all samples had deep and non-uniform grooves except for the T6 heat-treated alloy, which exhibits shallow and uniform grooves. Improvement of wear resistance of alloy was due to the result of finer Si particles as observed from Figure 10 (d) compared to (c) with CoF value of 0.4299 and 0.4231, respectively.



(a) (b)

(c) (d)

Fig. 10. Deformation through dry sliding of LM4 Al Alloy (a) As-cast, (b) CS casted, (c) Thixoformed, and (d) T6-thixoformed sample

4. Conclusions

This study shows that thixoformed LM4 Al alloy with optimized T6 heat treatment parameters of ST at 530 °C for 30 min, 2 hours of ageing at 180 °C attain better tensile strength, surface hardness, and CoF than the as-thixoformed and as-cast alloys. CS casting at 660 °C pouring temperature, 400mm slope length and 45 ° tilt angle produce the best billet feedstock for thixoforming as primary the irregular dendritic arms are broken and homogenously nucleate. Well-defined and near globular α-Al microstructure as secondary dendritic arms are broken is exhibited after thixoforming. T6 heat treatment adds enhancement on the already improved properties of LM4 alloy due to thixoforming process.

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