中國機械工程學刊第四十一卷第五期第 663~670 頁(民國一百零九年)
Journal of the Chinese Society of Mechanical Engineers, Vol.41, No.5, pp 663~670 (2020)

Studies on Microstructural and Tensile Behavior of Aluminium Metal Matrix Composites with Addition of SiC_p and Coconut Shell Ash by Squeeze Casting Method

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Keywords: LM24 aluminium alloy, silicon carbide, coconut shell ash, squeeze casting, Taguchi method, genetic algorithm

ABSTRACT

This experimental study focuses on processing of hybrid metal matrix (LM24-SiCp-coconut shell ash) composite for making castings through squeeze casting process. The primary objective was to analyze the influence of the process parameters namely reinforcement percentage, pouring temperature, squeeze pressure and mould temperature on response. Samples were cast for each experimental condition based on L9(34) orthogonal array. From ANOVA, it was observed that reinforcement percentage and squeeze pressure were the process parameters making a noticeable improvement in tensile strength. Scanning electron microscopy studies were carried on the fractured tensile test specimen to analyze the fracture mechanism.

INTRODUCTION

The process of selecting the appropriate materials for the engineering applications becomes a lot of tedious because the materials library keeps on updating with the latest innovations in materials development. World market pushes the research activities within the direction of composite materials instead of unreinforced alloys. Composite materials, especially Metal Matrix Composites (MMCs) are highly capable of replacing current monolithic materials for the specific engineering applications viz. aerospace, automobile, and marine improved wear resistance, superior corrosion resistance, improved tensile strength, toughness, and impact strength etc. In particular, aluminum based MMCs are gaining much attention among others for the reasons of compatibility with all kind of hard as well as soft reinforcements (Shetty et al., 2009; Surappa, 2003; Ibrahim et al., 1991; Adalarasan and Sundaram, 2015).

Generally, Metal Matrix composites (MMCs) are processed either by solid state processes (powder metallurgy) or liquid state processes (casting route). Squeeze casting is an emerging metal forming process that has been widely employed for the production of light metal alloy components with near net shaping (Yue, 1997; Abou El-khair, 2005). The applied pressure during solidification of the melt activated multidirectional feeding mechanisms throughout die cavity and hindered the formation of porosities caused by both gas and shrinkage (Rolland et al., 1993). The applied pressure is involved in microstructure grain refinement of squeeze cast aluminium alloys such as AC8A, LM6, LM13 and A356 (Lee et al., 2000; Vijian and Arunachalam, 2006; Maleki et al., 2006). Yue et al. (1997) observed when processing AA7010 wrought aluminium alloy through squeeze casting that high pouring temperature and high pressure were undesirable as they led to poor tensile property. Kim et al (1998) reported that squeeze cast 7050 wrought aluminium alloy parts exhibited superior quality than gravity die cast parts. Many researchers investigated the effects of various casting parameters on mechanical properties of squeeze cast aluminium alloys such as Al-Cu based alloy, Al-Zn-Mg-Cu alloy, Al-13.5%Si alloy, EN-AB46000, LM25, Al 2124, Al-7Si-0.7Mg and Al-7%wt Si (Gokhale and Patel, 2005; Yue, 1997; Zhang et al., 2007; Fan et al., 2010). and reported that mechanical properties were improved with the application of squeeze pressure. The optimum squeeze casting condition exhibited a significant improvement in mechanical properties of aluminium alloys like LM24, AC2A, A2017, AlSi7Mg, etc. (Senthil and Amirthagadeswaran, 2012, 2014; Manjunath Patel et al., 2016; Rajagopal, 1981). Ghomashchi and Vikhrov (2000) insisted for the

Paper Received August, 2019. Revised September, 2020. Accepted October, 2020. Author for Correspondence: M.Arulraj.

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further developments in squeeze casting process to produce complex shaped and thin walled alloy components.

Reinforcements of agro waste materials are offering attractive features like low-cost, low-density and less environmental pollution (Ravikumar et al., 2014; Manjunath Patel et al., 2016). The utilization of naturally available agro-waste particulates has become a very good reinforcement constituent due to the ease of availability. Therefore, many researchers conducted extensive studies for the enhancement of Mechanical properties of AMC's on several natural wastes from agriculture (Manjunath Patel et al., 2015; Alaneme et al., 2013). The most widely renowned agro-wastes are groundnut shell, cow horn, corn cob ash, coconut shell and bagasse. Though only limited literature was reported for the coconut shell ash (CSA) particles. The lack of literature for the use of coconut shell ash MMCs, especially for improving the primary mechanical properties motivated the authors to focus in the direction of developing high performance aluminium matrix composites.

Table 1. Chemical composition of LM24

Element	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Al
JIS (wt %)	7.5-9.5	≤ 3	3.0-4.0	≤ 0.5	≤ 0.3	≤ 0.5	≤0.5	≤ 3	Rest
Ingot (wt%)	7.848	0.785	3.433	0.14	0.15	0.025	0.049	1.334	86

MMC manufacturing could be performed using any one of a solid, liquid and vapor methods. Solid state methods includes powder metallurgy, diffusion bonding etc. whereas liquid state methods includes electroplating and electroforming, stir casting, pressure infiltration, squeeze casting. Physical vapor deposition is also a method for the manufacturing of MMCs in vapour method. Among these, stir casting process and squeeze casting methods are popular for processing Al-Si alloy composites in the production of castings by means of high-level pressure along with certain process parameter settings. Therefore, this study reports the microstructural behaviour and effect of squeeze casting process parameters on LM24-SiC_p-coconut shell ash composite while attempting for the improvement of tensile strength of the MMCs using coconut shell ash reinforcement. Results exhibit a definite contribution of coconut shell ash in the enhancement of tensile strength of the aluminium composites. The chemical composition of LM24 as per British Standard is given in Table 1. LM24 alloy is widely used for producing components such as piston, cylinder, connecting rod, etc (Singh et al., 2014). Hybrid (LM24-SiC_p-coconut shell ash) composite was processed for making casting through squeeze casting technique and optimization techniques were employed for finding optimum parametric condition for improving tensile strength of the castings in this study.

EXPERIMENTS AND TESTS

Squeeze Casting Experimental Setup

The experimental setup shown in Fig. 1 bottom pouring type stir casting furnace with squeeze casting setup manufactured by Swam equip factory consists of bottom pouring electric furnace, preheaters, split die set, punch and hydraulic circuit. The furnace capable of heating upto 1200°C was used to melt the metal at the desired temperature. A pathway with a preheater arrangement of 400°C capacity is inbuilt in this setup to maintain the fluidity of the molten metal during transferring from the furnace to the die cavity. SG400 spheroidal graphite iron (die material), H13 hot die steel (die insert material), EN8 alloy steel (punch material) and DYCOTE D140 (die coat material) were used. A permanent split die set specially made of H13 die steel and core of mild steel were designed for making hollow cylindrical sample of 50 mm outer diameter and 200 mm height. A preheater of 500°C capacity with thermocouple arrangement was used to preheat the die which enables uniform heating.

The inner sides of the stainless-steel crucible and die cavity were coated with graphite suspension. LM24 ingots were charged into the crucible in the furnace and the required temperature was maintained. The molten metal was degassed fully using hexachloroethane tablets to remove the entrapped gases and other impurities present. The molten slurry was transferred from the bottom pouring furnace into the preheated die through the preheated pathway which connects the melting furnace with the die. A hydraulic press of 40-ton capacity was used to apply squeeze load on the molten metal by means of a punch which is an integral part of the hydraulic unit. Squeeze pressure was applied on the melt through the punch and maintained until solidification was completed. Punch was then moved up and the casting was separated from the die assembly.

The reinforcement less than 2.5wt% did not show any appreciable improvement in impact strength and the reinforcement greater than 7.5wt% led to agglomerations in the castings. So, the addition of reinforcement was varied from 2.5wt% to 7.5wt%. Pouring temperature of 675ºC was required for effective filling of molten metal into the mould cavity. When the melt temperature was raised to above 725ºC, gases evolved from die coating were entrapped in the melt, leading to pin holes. The mould assembly was designed to withstand maximum squeeze load of 40 ton. It was observed that there was an existence of micro pores in the castings made at squeeze load of 20 ton (50 MPa). Hence, the bounds for all these process parameters were set as follows.

Reinforcement percentage, A (wt. %): $2.5 \leq A \leq 7.5$
Pouring temperature, B (ºC) : $675 \leq B \leq 725$
Squeeze pressure, C (MPa) : $50 \leq C \leq 150$

Mould temperature, D (ºC) : $200 \leq D \leq 300$

Fig. 1. Squeeze casting experimental set-up

All process parameters were fixed at three levels within the above bounds to conduct experiments based on the L9 $(3)^4$ orthogonal array. The details of squeeze casting process parameters and their levels are given in Table 2. For each experimental condition, the casting samples were cast and are shown in Fig. 2. The universal testing machine KUT-40 model was employed for performing the tensile test on the specimens. The tensile test specimens were prepared for each experimental condition as per the E8M-04 ASTM standard and it is shown in Fig. 3.

Fig. 2. Squeeze casting samples

Fig. 3. Tensile test specimens

Table 2. Squeeze casting process parameters and their respective levels

Process Parameter	Notation	Level		
		1	2	3
Reinforcement (wt. %)	A	2.5%CSA +7.5% SiC_p	5%CSA+ 5% SiC_p	7.5%CSA+ 2.5% SiC_p
Pouring temperature (°C)	B	675	700	725
Squeeze pressure (MPa)	C	50	100	150
Mould temperature (°C)	D	200	250	300

RESULTS AND DISCUSSION

Taguchi method

S/N ratio response

The tensile strength was treated as an output response with the category of quality characteristics “larger-the-better”. The S/N ratio for this response was estimated by using Equation (1) for each experimental condition and their values are given in Table 3.

$$S/N(\mathrm{dB}) = -10\log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{R_i^2}\right) \qquad (1)$$

where i = 1,2,…, n (here n = 4) and R_i is the response value for an experimental condition. Mean value ($\overline{Y}$) of S/N ratios was also calculated using Equation (2) and is given in Table 3.

$$\text{Mean}, \overline{Y} = \frac{1}{N}\left(\sum_{j=1}^{N} Y_j\right) \qquad (2)$$

where j = 1,2… N (here N = 9) and Y_j is S/N ratio for j^{th} parametric setting.

Table 3. Experimental observations and S/N ratio for hardness

Ex. No.	Parameters and their levels				Tensile Strength (MPa)					S/N Ratio (dB)
	A	B	C	D	R_1	R_2	R_3	R_4	R_{avg}	
1	1	1	1	1	321	342	339	326	332	50.4227
2	1	2	2	2	341	350	351	342	346	50.7815
3	1	3	3	3	385	385	382	388	385	51.7766
4	2	1	2	3	340	350	354	356	350	50.8813
5	2	2	3	1	360	361	364	363	362	51.1741
6	2	3	1	2	318	328	324	326	324	50.2109
7	3	1	3	2	344	352	350	362	352	50.9308
8	3	2	1	3	308	310	309	313	310	49.8272
9	3	3	2	1	316	316	320	320	318	50.0485
									$\overline{Y}$	50.6726

In order to find optimum level of the process parameters, average S/N ratio response was estimated for every level of the parameters and the corresponding details are given in Table 4. Based on the highest value of S/N ratio, an optimum level for each parameter (A: 1stlevel; B: 1st level; C: 3rdlevel; D: 3rdlevel) was noted. Thus, the optimum parametric setting $A_1B_1C_3D_3$ (reinforcement percentage: 90%LM24+2.5% CSA+7.5% SiC_p, pouring temperature:675ºC, squeeze pressure:150MPa and mould temperature: 300ºC) was obtained for the output response.

Table 4. Average S/N ratio response table

	A	B	C	D
Level1	50.9936	50.7449	50.1536	50.5484
Level2	50.7554	50.5942	50.5704	50.6411
level3	50.2688	50.6786	51.2938	50.8284
Max-Min	0.7248	0.1507	1.1402	0.280
Rank	2	4	1	3
Optimum	A1	B1	C3	D3

The Main Effect plot of the response graph shown in Fig. 4 described the variation of each process control parameter on the output response of the squeeze casting process. Fig. 6.1-6.3 shows the effect of different reinforcement wt. % (2.5 wt.% CSA +7.5 wt.% SiC_p, 5 wt.% CSA +5 wt.% SiC_p and 7.5 wt.% CSA+2.5 wt.% SiC_p), squeeze pressure of (50, 100 and 150 MPa), pouring temperatures (675ºC, 700ºC, 725ºC) and mould temperature (200ºC, 250ºC, 300ºC).

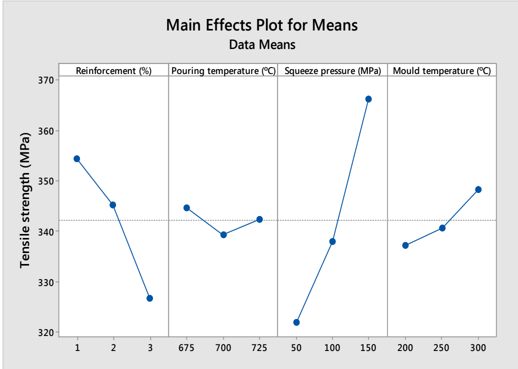

Fig. 4. Main Effect plot of the response - Tensile strength

It is observed that the maximum results were obtained for 2.5 wt.% CSA +7.5 wt.% SiC_p. It is expected that due to the thermal coefficient of mismatch and strong internal stresses would deform at the particle-matrix interface, it would provide better load transfer from the matrix to the particle resulting in higher strength. Beyond 2.5 wt.% CSA and 7.5 wt.% SiC_p the tensile strength and impact strength may decrease due to the formation of agglomeration, micro porosity and shrinkage (Ghomashchi and Vikhroy., 2000). Pouring temperature has a significant influence on heat transfer and distribution of particles and it directly affects mechanical properties of the composite. At 675ºC the mechanical properties decreased due to the fact that the lower die surface temperatures prevent effective filling of the die and cause premature solidification of the melt. The higher die temperature about 725ºC causes an increase in solidification time. During this period, particle settling could be taken place (Singh et al., 2014). These factors result in the formation of particle clusters, shrinkage, and porosity in the casted composite material. The cluster regions and the pores act as the stress-initiating zone in the composite while loading and it decreases the tensile strength and impact strength. Further, uneven distribution due to agglomeration reduces the hardness of the composite. All these negative effects can be annihilated at the pouring temperature of 700ºC for this volume and shape of the composites that produces the maximum results.

When applied pressure increased from 50 MPa to 150 MPa, the results indicated a drastic improvement in hardness, tensile strength, and impact strength values. This may be attributed to the refinement in the microstructure due to the high-pressure level. The maximum values of mechanical properties were obtained for the maximum squeeze pressure of 150 MPa for this volume and shape of the composites. Improvement in hardness may be attributed to the fact that (i) the harder SiC_p and CSA particles reinforced in the softer aluminium matrix resists plastic deformation (ii) the externally applied pressure increases the density of the composite thereby increasing its tensile strength. Mould temperature has a significant influence on heat transfer and distribution of particles and it directly affects mechanical properties of the composite. At 200ºC the mechanical properties decreased due to the fact that the lower die surface temperatures prevent effective filling of the die and also causes premature solidification of the melt. The higher die temperature about 300ºC causes an increase in solidification time (Manjunath Patel et al., 2016).

Analysis of variance (ANOVA)

The purpose of analysis of variance (ANOVA) is to investigate which process parameters that significantly affect the quality characteristic. ANOVA determines the optimum combination of process parameters more accurately by investigating their relative importance among them. It was performed on signal-to-noise ratios to find the relative significance of the process control parameters and their contribution in the process performance. The following terms were calculated by using Equations (3-10) and their values are given in Table 5.

(i) Sum of squares due to mean, $SS_m = N\overline{Y}^2$ (3)

(ii) Sum of squares due to parameter A,

$$SS_A = n_{A1} \times \overline{A_1}^2 + n_{A2} \times \overline{A_2}^2 + n_{A3} \times \overline{A_3}^2 - SS_m \quad (4)$$

Similarly, sum of squares due to parameters B, C and D were calculated. Sum of squares due to parameters B and D were found to be very less in this study. Therefore, their effects on the output response were assumed to be insignificant and considered as an error (pooled error). Sum of squares due to pooled error was also calculated as follows.

$$SS_{\text{pooled error}} = SS_B + SS_D \quad (5)$$

(iii) Total sum of squares,

$$TSS = SS_A + SS_B + SS_C + SS_D \quad (6)$$

(iv) Degree of freedom for parameter,
$DOF_{\text{parameter}}$ = Number of parameter levels – 1
Degree of freedom for pooled error,
$DOF_{\text{pooled error}} = DOF_B + DOF_D$

(v) Mean sum of squares due to parameter A,

$$MSS_A = \frac{SS_A}{DOF_A} \quad (7)$$

Likewise, mean sum of squares for all other parameters and pooled error were calculated.

(vi) F ratio for parameter A,

$$F_A = \frac{MSS_A}{MSS_{\text{pooled error}}} \quad (8)$$

Similarly, F ratio was calculated for parameter C. The calculated F ratio for parameters A and C was found to be greater than the F distribution value ($F_{1,4}$= 7.71 at 5% level of significance). Therefore, the parameters A and C were confirmed as significant parameters in this study.

(vii) Pure sum of squares due to parameter A,

$$PSS_A = MSS_A - DOF_A \times MSS_{\text{pooled error}} \quad (9)$$

(viii) Percentage contribution of parameter A,

$$PC_A = \frac{PSS_A}{TSS} \times 100\% \quad (10)$$

Similarly, pure sum of squares and percentage contribution of parameters C and pooled error were calculated. The percentage contribution of pooled error was noted to be less than 5% in this study. The percentage contribution of all significant parameters is clearly shown in Fig. 5. Taguchi method was used to find better level of process parameters from among the set levels. With the intention of tuning the parameter setting between the set levels, metaheuristic technique namely genetic algorithm was also used in this study.

Table 5. ANOVA table

Source	Pool	SS	DOF	MSS	F ratio	PSS	PC (%)
A		0.8194	2	0.4097	10.42	0.7801	33.19
B	Yes	0.0043	2	0.00215			
C		1.9796	2	0.9981	25.39	1.9797	58.89
D	Yes	0.1529	2	0.07645			
Pooled Error		0.1572	4	0.0393		0.2144	7.92
TSS		2.9742				2.9742	100

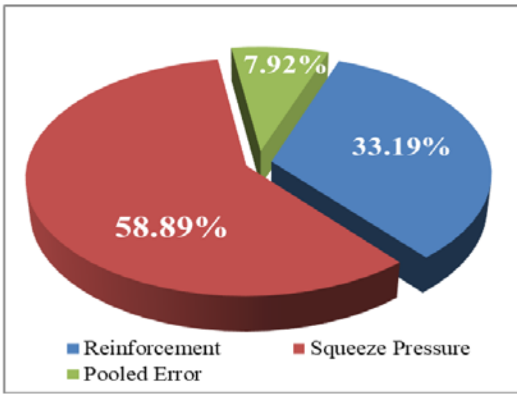

Fig. 5. Percentage contributions of significant parameters

Analysis of Microstructure

Fig. 6 shows the microstructure of the HAMCs solidified under 150 MPa squeeze pressure, 700ºC pouring temperature, 250ºC mould temperature, and varying weight % reinforcement (2.5 wt.% CSA +7.5 wt.% SiC_p). The castings obtained with the 2.5 wt. % CSA +7.5 wt.% SiC_p of reinforcement for the squeeze casting condition, showed better grain refinement in the microstructure shown in Fig. 5. Due to the high level squeeze pressure, heat transfer increased considerably between the melt and the mould, which led to an increase in solidification rate. High heat transfer or cooling was the reason for good grain refinement in the microstructure (Arulraj and Palani, 2018). It is observed that the minimum wear rate was obtained for 2.5 wt.% CSA +7.5 wt.% SiC_p. It is expected that due to the thermal coefficient of mismatch and strong internal stresses would deform at the particle-matrix interface, it would provide better load transfer from the matrix to the particle resulting in lower wear rate.

Fig. 7 shows the microstructure of the HAMCs solidified under 100 MPa squeeze pressure, 700ºC pouring temperature, 250ºC mould temperature, and varying weight % reinforcement (2.5 wt.% CSA +7.5 wt.% SiC_p). The castings obtained with the 2.5 wt. % CSA +7.5 wt.% SiC_p of reinforcement for the squeeze casting condition showed formation of agglomeration in the microstructure when the squeeze pressure applied was 100 MPa. Fig. 6 shows the microstructure of the HAMCs solidified under 50 MPa squeeze pressure, 700ºC pouring temperature, 250ºC mould temperature, and varying weight % reinforcement (2.5 wt.% CSA +7.5 wt.% SiC_p). Porosities and segregations were observed in the castings for applied pressure of 50 MPa.

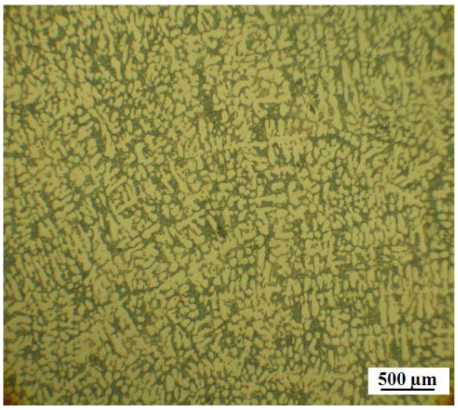

Fig. 6. Microstructure for HAMCs at 2.5 wt.% CSA + 7.5 wt.% SiC_p (squeeze pressure of 150 MPa, pouring temperature of 700ºC and mould temperature of 250°C are fixed)

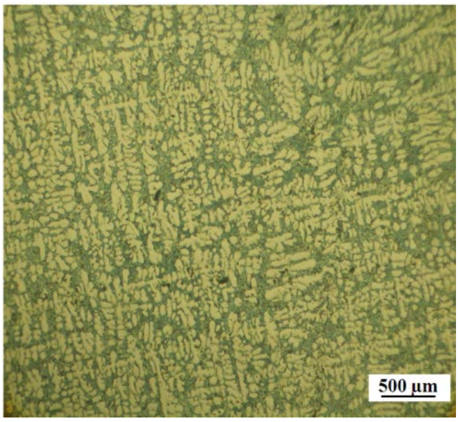

Fig. 7. Microstructure for HAMCs at 2.5 wt.% CSA + 7.5 wt.% SiC_p (squeeze pressure of 100 MPa, pouring temperature of a 700ºC and mould temperature of 250°C are fixed)

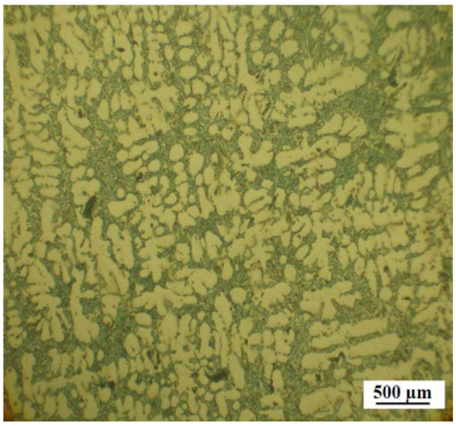

Fig. 8. Microstructure for HAMCs at 2.5 wt.% CSA + 7.5 wt.% SiC_p (squeeze pressure of 50 MPa, pouring temperature of a 700ºC and mould temperature of 250°C are fixed)

Tensile Fracture Surface Analysis

The SEM of the fractured tensile test specimens is shown in Fig. 9-11. Presence of hard particles transfers the applied load from the aluminium matrix to the reinforcement and increases the resistance to plastic deformation of composites. This takes place due to the thermal mismatch between the aluminium matrix having a high coefficient of thermal expansion and the reinforcement particles with a low coefficient of thermal expansion. A thermal mismatch generates thermal stress in the composites and leads to the formation of dislocation at the interface. Presence of the hard SiC_p and CSA particles in the ductile aluminium matrix initiates the formation of micro cracks. Lack of interfacial strength between Al/SiC_p/CSA may be due to partial wetting at the interface. Ductile failure in the form of delamination is observed in composites having a composition of 2.5 wt.% CSA and 7.5 wt.% SiC_p in Fig. 9. Presence of SiC_p particles reduces the formation of dimples long the surface indicating a decrease in elastic deformation of the composites. Micro cracks and fractured SiC_p and CSA particles were observed in Fig. 10 indicating an increase in the brittle nature of the composites. With further addition of CSA in Fig. 11 debonding and particle crack are evident along the surface influencing an increase in brittility of the composite. Increase in reinforcement particles increases the stress concentration points at the interface thereby decreasing the strength and elongation of the composites. The impurities present in the reinforcement increases the porosity of composites and decrease the strength of composites.

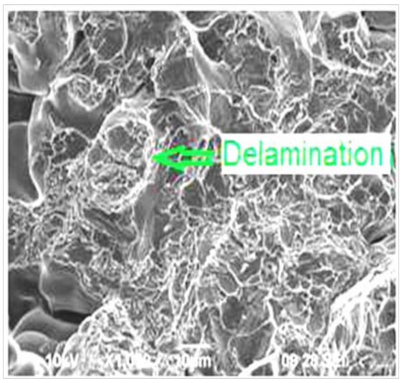

Fig. 9. SEM of the fractured Al/2.5 wt.% CSA/7.5 wt.% SiC_p composites after tensile test

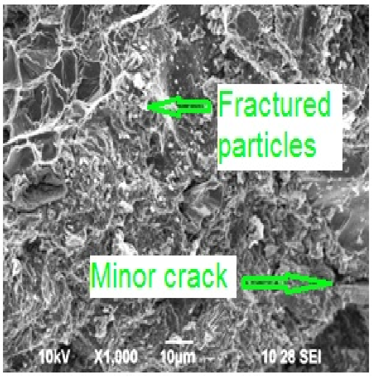

Fig. 10. SEM of the fractured Al/5 wt.% CSA/5 wt.% SiC_p composites after tensile test

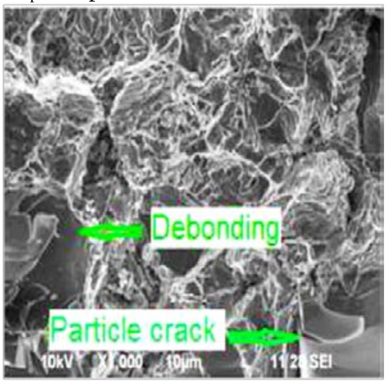

Fig. 11. SEM of the fractured Al/7.5 wt.% CSA/2.5 wt.% SiC_p composites after tensile test

CONCLUSION

The following are the conclusions drawn based on the parametric optimization for improving tensile strength of squeeze cast hybrid (LM24-SiC_p-coconut shell ash) composite castings.

i) From the ANOVA, squeeze pressure and reinforcement weight fraction were identified as significant process control parameters in this study.

ii) From the percentage contribution analysis, it was noted that squeeze pressure was the most important parameter over the control of squeeze casting process.

iii) The optimum squeeze casting conditions were obtained through Taguchi method as reinforcement percentage: 90% LM24 + 2.5 wt.% CSA + 7.5 wt.% SiCp, pouring temperature: 700ºC, squeeze pressure: 150 MPa and mould temperature: 250ºC.

iii) The castings obtained for the optimum squeeze casting condition showed better grain refinement in the microstructure and nearly 25% improvement in tensile strength than the base alloy.

iv) The tensile fracture surface analysis of composites concluded that the fracture surface exhibited a combined fracture mode of brittle and ductile fracture. It was observed that the fracture occurred due to particle cracking, interface debonding and deformation constraint in the matrix.

Nomenclature

SiC_p : Silicon Carbide Particle
CSA : Coconut shell ash
DOF : Degree of freedom
MSS : Mean sum square
PSS : Pure sum of squares
PC : Percentage contribution
SS : Sum of squares
TSS : Total sum of squares

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