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OPTIMISATION OF STIR CAST PROCESS PARAMETER OF CARBON NANOTUBE REINFORCED ALUMINIUM ALLOY METAL MATRIX COMPOSITE USING RESPONSE SURFACE METHODOLOGY (RSM)

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Abstract

The paper presents the results of experimental investigations on tensile strength and optimization of stir casting process parameters of carbon nanotube (CNT) reinforced aluminium 2024 metal matrix composite using Response surface methodology (RSM). The effect of casting parameters such as percentage of reinforcement fraction, stirring time, stirring speed and casting temperature of Al 2024 metal matrix composite manufactured through stir cast route method was studied. An empirical model was developed for predicting the tensile strength of CNT reinforced aluminium 2024 metal matrix composite. Adequacy of the developed model was tested using ANOVA and found to have excellent predictive capacity as indicated by the values of R^2 of 0.8904. The optimum process conditions were established to be 1.93 % reinforcement fraction, 433.5 rpm stirring speed, 690 °C processing temperature and 150sec stirring time. At this condition, an optimal increase of 76% in tensile strength of the composite was observed using desirability function approach.

Keywords: Al2024, carbon nanotube, stir casting, Anova, RSM.

1.0 INTRODUCTION

Lightweight, high strength materials have been required since the invention of the airplane. Increasing the strength and stiffness of a material, the dimensions, and consequently, the mass, of the material required for a certain load bearing purpose is reduced. This leads to several advantages in the case of aircraft and automobiles such as increase in payload and improvement of the fuel efficiency. With global oil resources on a decline, increase in the fuel efficiency of engines has become highly desirable. Metals and alloys resulted are deficient in providing both strength and stiffness and therefore the development of alternative materials has become necessary. Metal matrix composites (MMCs) could be a solution, with lightweight and ductility provided by the metal and the strength and stiffness by the reinforcement that is either a ceramic or high stiffness metal based particulate or fiber (Bakshi *et al.*,2010).

Particulate Aluminum Metal Matrix Composite (PAMMCs) was stirred by S. Ray first time in 1968, where alumina (Al_2O_3) particles were introduced into molten aluminium by mechanical stirring. The major disadvantage of this process is agglomeration of particles during fabrication process (Girot *et al.*, 1987; Harnby *et al.*, 1985; Kala *et al.*, 2014). Stir casting is an economical, effortless and most commercially adopted technique (Surappa *et al.*, 1981), and it is known as vortex technique (Nagaral *et al.*, 2015).

Powder metallurgy which is the most preferred method of MMCs preparation due to its low processing temperature employed in mixing the powder is quite costly and not applicable to complex shapes. Other manufacturing techniques

such as squeeze, melt impregnation and spray casting methods are also constrained by certain disadvantages which include high cost as well as restricted size and limited shapes of the final product. Many authors fabricated PAMMCs with different reinforcement through stir casting route and tested the mechanical properties. Thomas and Parameshwaran, (2014) fabricated the Aluminium LM6 reinforced with 15% SiC stirred through manual mixing, existed mechanism of stir casting and modified mechanism which is stirrer with two blade assemblies and holes in the feeder along with rotation of feeder at 800 rpm. The experimental result of existing mechanism were compared with modified mechanism. Investigations revealed that the percentage elongation was increased by 34%, buckling load was increased by 5.45% and increased by BHN value of 4.3 % (Thomas *et al.*, 2014).

The present study takes advantage of stir casting technique which is proven to be more viable owing to its simplicity, flexibility and ability to produce large sized and complex shaped component. It is also attractive because it allows a conventional processing route to be used and hence minimizes the final cost of the product.

Response surface methodology (RSM) is applied in studying the relationships between several explanatory factors and one or more response variables, it is a popular approach on optimization of the input parameters models based on simulation and experimental observations. These models need to be calculated statistically for their suitability, and then they can be utilised for an optimisation of the initial model. It also measures the relationships between the controllable input parameters and the obtained response surfaces. The input parameters are sometimes called independent variables, and the performance measure is the response. Response surface methodology analysis is computationally much less expensive than a solution using the traditional method. By using the analytical model, the objective function problem can be easily and timely solved. (Aggawal *et al.*, 2008).

2.0 MATERIALS AND METHODS

2.1 Materials

The matrix material used in this study is Al 2024 obtained from Scientific Equipment Development Institute, Minna, Nigeria. The Multiwalled Carbon Nanotubes which was 16-50 nm diameter and approximately 33 μm in length (Plate 1) was obtained from Center of Genetic Engineering and Biotechnology, Federal University of Technology Minna, Nigeria.

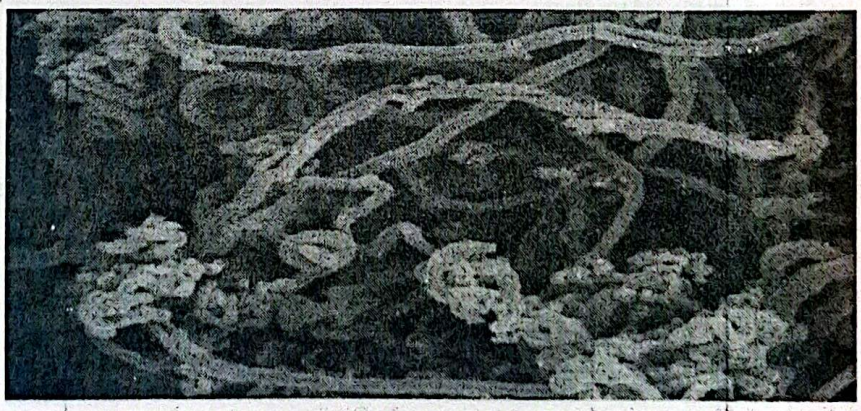
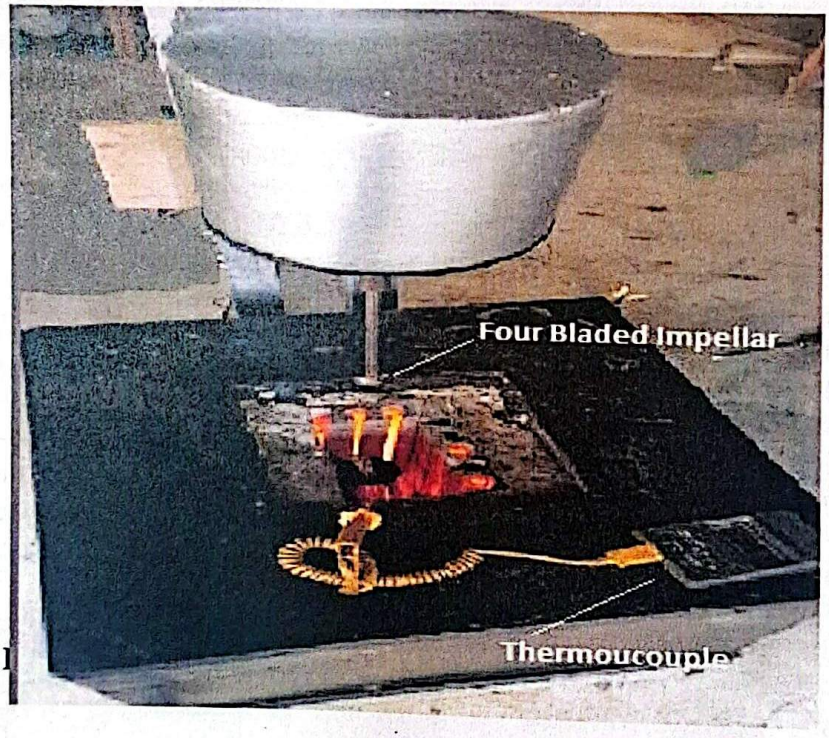


Plate 1: SEM Image of Carbon Nanotubes

2.2 Experimental Procedure

The experiment commenced by melting 200g of Al 4wt% Cu alloy ingot in the cylindrical stainless steel crucible in the electric resistance furnace (as shown in Figure 1). The CCD experimental arrangement generated through Design Expert software as presented in Table 1. Once the processing temperature of 700 °C of the first run was attained, the mechanical stirrer was switched to a speed of 600 rpm and lowered into the molten metal. A safe clearance of about 50 mm was ensured between the impeller and the bottom of the crucible. MWNT (1.5wt%) was preheated at 200 °C for 30 min and was gradually introduced into the molten metal using injection funnel while stirring continued for 120 secs. . The stirrer was raised at the end of the mixing and the mixture was poured into the prepared sand mould immediately. Digital thermocouple was used after the stirring to ensure that precise processing temperature was attained.



The detail of the central composite design matrix for composite production is presented in Table 1.

Table 1: Factors and their Level in CCD Experimental Design Plan

Factors		Levels				
		-2	-1	0	1	2
Reinforcement Fraction (wt %)	A	0.5	1	1.5	2	2.5
Stirring Speed (rpm)	B	200	300	400	500	600
Processing Temperature (^{0}C)	C	680	690	700	710	720
Stirring Time (Sec)	D	60	90	120	150	180

Tensile test was carried out on the prepared specimens as per ASTM standard E8 in order to evaluate the tensile strength of the composites.

2.2.1Tensile Test

Tensile testing was conducted using Dog-bone tensile test samples which were machined according to ASTM E8 standard. The testing was performed using 10kN Monsato Tensometer at a strain rate of 5 X 10^4 s^{-1}. The tensometer is equipped with data acquisition system which supplies the stress - strain curve during tensile test at room temperature. The test involves taking a sample of fixed cross-section area, and then pulling it by gradually increasing the force at a speed of 5 µm/s until the sample changes shape or breaks. The tensile test was carried out for all specimens respectively.

2.2.2 SEM of Produced Composite

SEM observation of the produced aluminum grains in composite was performed. In preparation for SEM imaging, the composite was cut into a half-moon shape and etched using nitric acid. SEM samples were prepared by finishing the polished surface of the cross section of composites using a cross-section polisher.

Table 2: Central Composite Design Matrix for Composite Production

Run	Reinforcement Fraction (%)	Stirring Speed (rpm)	Processing Temperature (°C)	Stirring Time (Secs)	Tensile Strength N/mm^2
1	1.5	600	700	120	98
2	1	500	690	150	113
3	2	500	710	90	110
4	2	300	690	150	110
5	2.5	400	700	120	130
6	1	300	690	90	100
7	1.5	400	700	120	134
8	1.5	400	700	60	120
9	1	300	690	150	111
10	1.5	400	700	120	135
11	0.5	400	700	120	104
12	2	500	710	150	114
13	1	300	710	150	100
14	2	500	690	150	158
15	2	300	710	90	100
16	1.5	400	700	120	135
17	1.5	400	700	180	139
18	1.5	400	680	120	126
19	1	500	710	150	105
20	1.5	400	700	120	133
21	1	500	690	90	130
22	1.5	400	700	120	125
23	2	300	710	150	102
24	1.5	400	700	120	132
25	1.5	200	700	120	100
26	1.5	400	720	120	129
27	1	300	710	90	100
28	2	300	690	90	104
29	2	500	690	90	143
30	1	500	710	90	104

2.2.3 Statistical Analysis of Process Parameters

Analysis of variance (ANOVA) was used in evaluating the statistical significance of the model equation. The results obtained showed that the regression is statistically significant at 95 % confidence level ($p< 0.05$) as indicated by Table 3. The model F-value of 4.039 and $p < 0.0055$ for composite production implied that the model was statistically significant. In this case, the model terms C, D, AD and CD are significant. The fitness of the model, the regression equation and R^2 were evaluated. The value of R^2 was 0.8904, is an indication that the model could explain 89.04% of the sample variability. The adjusted R^2 obtained was 0.8947 thereby indicating the significance of the model. The final regression equation for the model in coded terms is given as;

Tensile Strength = 132.33+5.42A+6.08B-5.33C+2.50 D+4.25AB-2.75AC+2AD-5.50BC-1BD-0.5CD - $4.75A^2$-$9.25B^2$-$2.12C^2$-$1.62D^2$

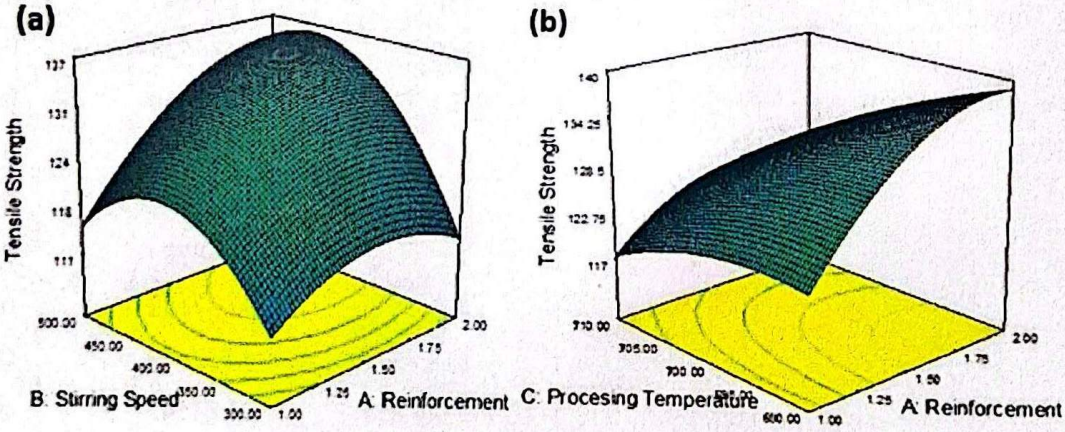
Table 3: Analysis of Variance on Tensile Strength of the Composite

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	6083.8	14	434.5571	4.039469	0.0055
A-Reinforcement	704.1667	1	704.1667	6.545652	0.0218
B-Stirring Speed	888.1667	1	888.1667	8.256042	0.0116
C-Processing Temperature	682.6667	1	682.6667	6.345796	0.0236
D-Stirring Time	150	1	150	1.39434	0.2561
AB	289	1	289	2.686428	0.1220
AC	121	1	121	1.124768	0.3057
AD	64	1	64	0.594918	0.4525
BC	484	1	484	4.49907	0.0510
BD	16	1	16	0.14873	0.7052
CD	4	1	4	0.037182	0.8497
A^2	618.8571	1	618.8571	5.752649	0.0299
B^2	2346.857	1	2346.857	21.81545	0.0003
C^2	123.8571	1	123.8571	1.151326	0.3002
D^2	72.42857	1	72.42857	0.673267	0.4248
Residual	1613.667	15	107.5778		
Lack of Fit	1542.333	10	154.2333	10.81075	1.0085
Pure Error	71.33333	5	14.26667		
Cor Total	7697.467	29			

The terms A, B, AD, BC, A^2 and C^2 are significant as such they have major impact on composite tensile strength. The terms C, AC, BC, with negative coefficient from Equation 1 all exhibits inverse proportionality behaviour with respect to the tensile strength of composite, while all terms with positive coefficient shows direct proportionality to the tensile strength.

2.2.4 Interactive Effect Plots

The surface plots of the responses are presented in Figure 2 (a-f) with the purpose of understanding the main and interaction effects of each factor. The tensile strength has been plotted as a function of the normalized independent variables. It has been indicated that the stirring speed has the most significant effect on the tensile strength. The three dimensional plot of Figure 2a shows that a combination of intermediate stirring speed and high reinforcement fraction favours tensile strength than other process conditions. Figure 2b shows the synergetic effect of processing temperature and high reinforcement fraction. The plot indicates that the reinforcement fraction has a significant effect on the composite tensile strength than processing temperature. Thus it can be seen that a combination of low processing temperature and high reinforcement fraction results in higher tensile strength. Thus increasing reinforcement fraction increases the tensile strength to a large extent while increase in processing temperature from 600 –710°C resulted to decrease in the yield. The relationship between processing temperature and stirring speed is shown in Figure 2c.



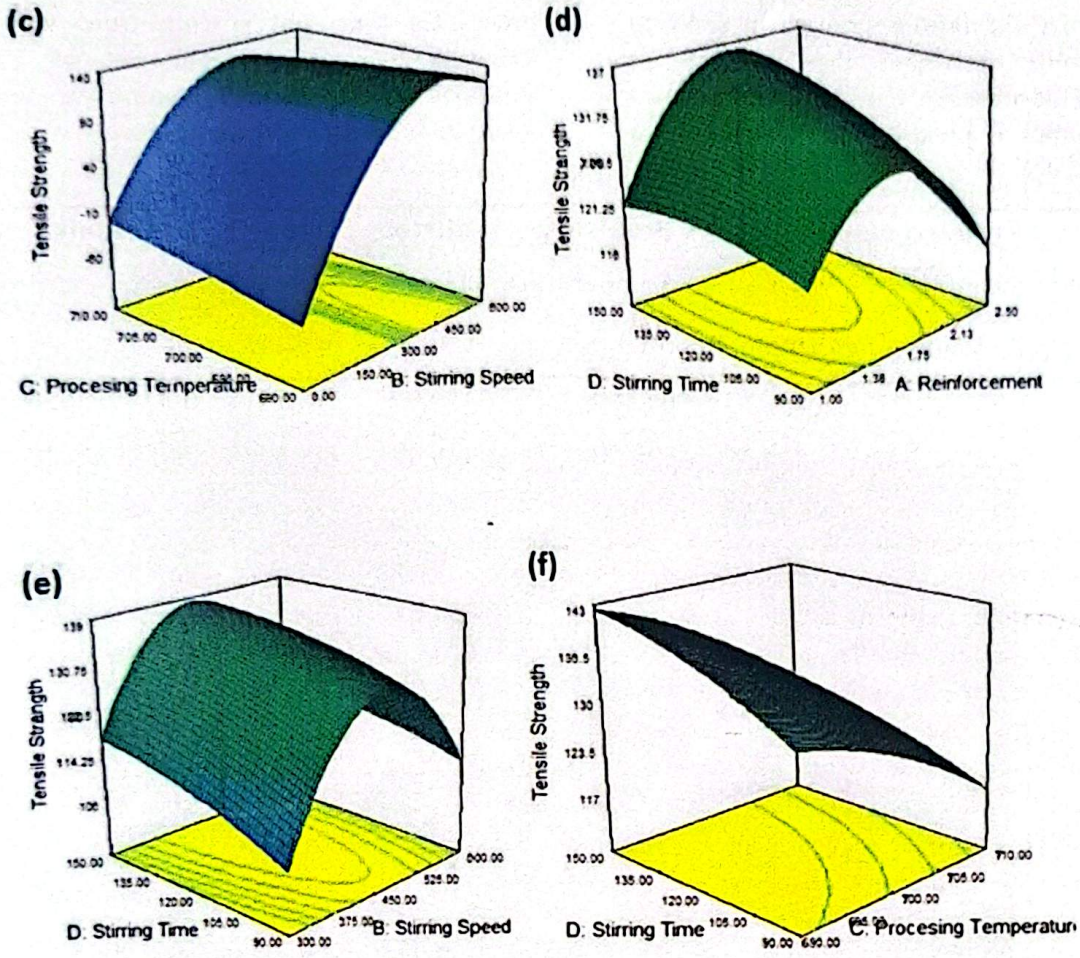


Figure 2: Interactive Effect Surface Plots of (a) Stirring Speed –Reinforcement Fraction (b) Processing Temperature-Reinforcement Fraction (c) Processing Temperature Stirring Speed (d) Stirring Time-Reinforcement Fraction(e) Stirring Time-Stirring Speed (f) Stirring Time-Processing Temperature on Tensile Strength (N/mm^2)

The curvature nature of the plot suggests that a significant interaction exits between the two terms. It shows that the combination of high processing temperature and stirring speed when use for composite production favors tensile strength. The resultant effect between the stirring time and reinforcement fraction rate is illustrated in Figure 2d. The result also shows that increasing the value of stirring time from 60 to 150 seconds while holding the reinforcement fraction at 2 wt% significantly influenced tensile strength. Similar trend was also observed between the stirring speed and stirring time in Figure 2e. It demonstrated that a combination of high stirring time and intermediate stirring speed resulted to the highest value of tensile strength. However, even at high stirring time the tensile strength reduces as the processing temperature increases from 690 to 710 °C as shown in Figure 2f.

2.2.5 Optimization of Stir Cast Process Parameters

Response optimization was carried out using desirability function in conjunction with response surface methodology. Various multi-characteristic models have

been developed. Goals were set to maximize the tensile strength in order to accurately determine their impact on overall desirability. The ranges and goals of input parameters which are reinforcement fraction, stirring speed, processing time and stirring time were assigned appropriately. The set of 21 solutions obtained for desirability are given in Table 4.

Table 4: **Set of Optimal Solutions for Desirability**

S/n	Reinforcement Fraction (%)	Stirring Speed (rpm)	Processing Temperature (oC)	Stirring Time (Secs)	Tensile Strength N/mm^2	Desirability
1	1.93	433.53	690.00	150.00	176.7014	0.874459526
2	1.93	434.25	690.00	150.00	176.6985	0.874427376
3	1.93	433.59	690.00	149.66	176.6980	0.874421835
4	1.92	433.59	690.00	149.71	176.6970	0.874411544
5	1.94	435.38	690.00	149.99	176.6963	0.874403576
6	1.93	435.71	690.00	150.00	176.6927	0.874363128
7	1.92	432.85	690.00	148.57	176.6829	0.874254280
8	1.93	428.19	690.00	149.84	176.6636	0.874040217
9	1.92	435.51	690.00	146.98	176.6601	0.874000971
10	1.92	435.03	690.00	146.44	176.6533	0.873925110
11	1.94	432.77	690.10	149.99	176.6513	0.873903631
12	1.93	436.33	690.00	145.39	176.6349	0.873720813
13	1.93	433.03	690.00	145.32	176.6274	0.873637885
14	1.96	439.26	690.00	147.91	176.6273	0.873636933
15	1.93	433.05	690.21	150.00	176.5979	0.873310511
16	1.94	439.90	690.00	143.62	176.5800	0.873110719
17	1.96	442.59	690.00	141.56	176.4847	0.872052656
18	1.88	441.47	690.00	144.38	176.4796	0.871995260
19	1.91	437.68	690.00	138.63	176.4599	0.871777131
20	1.82	433.92	690.00	144.19	176.2653	0.869614873
21	2.00	439.60	691.68	140.17	175.2158	0.857953470

The set of conditions possessing highest desirability value is selected as optimum condition for the desired responses. In this case, the optimal values of the process parameters are 1.93 wt % weight fraction, 433.5 rpm stirring speed, 690 °C processing temperature and 150 secs stirring time. Similar findings were also reported by Adebisi *et al* (2015).

The SEM micrograph of the produced composite is presented in Figure 3. Although it is difficult to view the MWNT in the composite under the SEM image, however the accompanied EDS confirms its presence. The nanosized MWNT addition from 2 wt% lead to the refining of grains, making a coaxial finer grain structure than the matrix alloy.

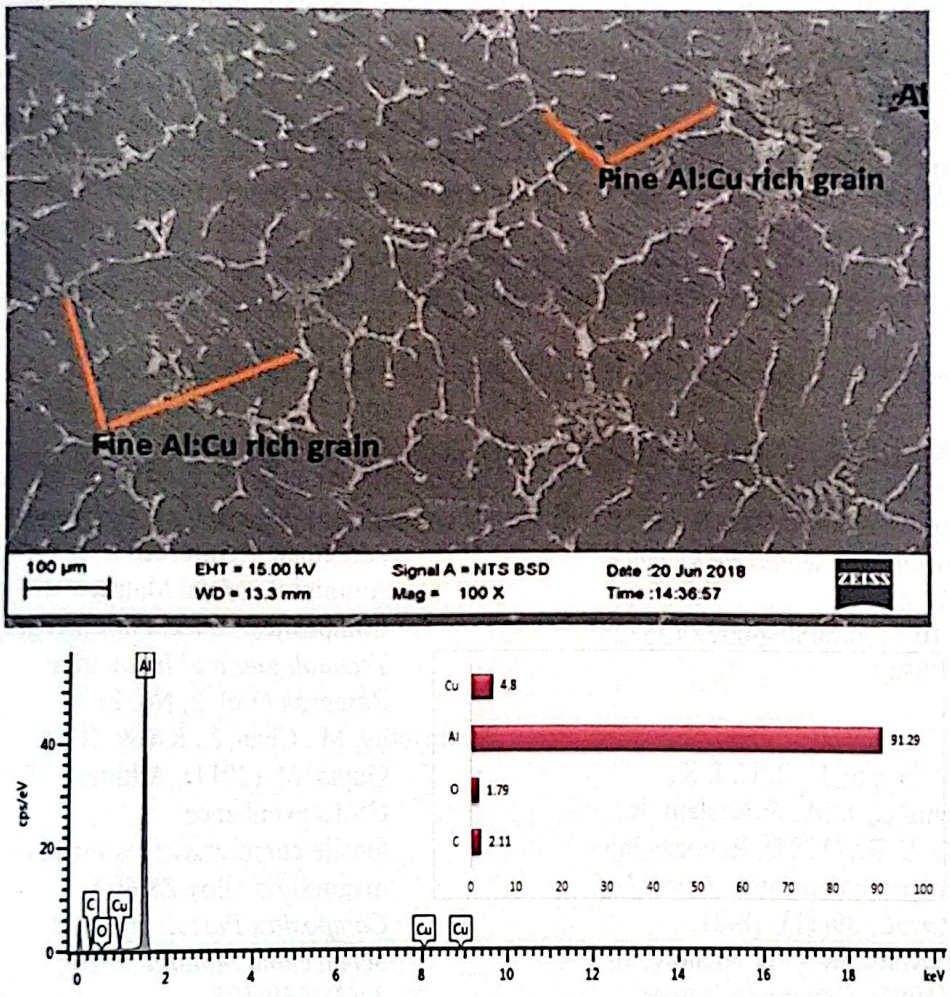


Figure 3: Scanning Electron Microscpe Image/ EDS of CNT Reinforced Aluminium Alloy

For nanocomposite materials, the grain size of the matrix depends on the particle size or the volume fraction of the particles. As the particle size decreases or volume fraction of nanoparticles increases, the grain size of the matrix decreases (Ezatpour *et al.,* 2014). This behavior is attributed to a higher incidence of grain boundary pinning which prevents grain growth. It has been observed that grain refinement occurs when a large amount of MWNT reinforcement is added to the matrix (Ceschini *et al.* 2013). Choi *et al.,* (2012) also reported the formation of ultrafine grained Al based MWNT composite. The well dispersed MWNT

was observed to form strong interface with the matrix by mechanical interlocking.
Aside grain refinement, other possible strengthening mechanisms resulting from MWNT addition that could lead to the enhancement of the mechanical properties are load transfer, the thermal mismatch and orowon looping effects. (Esawi *et al.*, 2010)

3. CONCLUSION

Stir cast process parameters resulted in the increase of 76% tensile strength. The optimum process conditions were established to be 1.93% reinforcement fraction, 433.5 rpm stirring speed, 690 °C processing temperature and 150sec stirring time.

The empirical models developed based on CCD and factorial designs have the potential to predict the catalyst and CNTs yield as well as composite responses within the design space. The adequacy of the developed models were tested using ANOVA and found to have excellent predictive capacity as indicated by the values of R^2 of 0.8904. Using such model a remarkable saving in time and cost has been obtained.
Micro structural observation of the cast composite revealed that more dispersed and smaller sized grain structures were formed in samples with the most enhanced mechanical properties. It is therefore established that the strength of the composite is influenced by the process parameters through modification of grain morphology.

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