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Synthesis and Characterization of Sic-Mg-Gr Nano Crystalline Reinforced Aluminium Matrix Composite and Analyis of Its Machinability Characteristics

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Abstract— A hybrid metal matrix composite containing a novel combination of two metals aluminium and magnesium with a ceramic silicon carbide has been synthesized. A sizable percentage of a lubricating material Graphite has also been added to this unique combination. ALUMINIUM is used as a base metal. Mechanical alloying technique has been adapted to synthesis the hybrid nano particles for reinforcement. The average particle size of the nano hybrid is 70nm after milling the materials for 12 hours. Nano particles together with aluminium as base materials are used to form a hybrid composite with varying propositions. The hybrid metal matrix composite has been analysed for its characteristics using SEM(scanning electron microscope) and XRD. Powder metallurgical process and stir casting are adopted to make pellets and to cast structures like plate and rod. The resultant metal matrix composite has been tested for its hardness and tensile strength. The values of hardness are measured in Vickers for pellets and cast materials. By the data derived from the various analysis and tests, it has been concluded that the plates cast using stir casting technique showed substantial hardness, density and tensile strength. The machinability aspects have been analysed after machining, the as cast plate in the Wire-cut Electrical Discharge machine. Taguchi method has been used to do the experimental design. The parameters have also been optimized using the ANOVA technique. The novel material with unique combination of metallic and ceramic constituents shows good mechanical characteristics and it is conductive to be machined using Wire cut EDM.

Keywords- *Hybrid composites, Mechanical alloying, SEM, XRD, ANOVA*

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I. INTRODUCTION

The advent of nanotechnology has given the researchers enough scope to find alternate combinations of materials for various applications aimed at enhanced performance. Nanocomposites have effectively been found as a suitable replacement for several existing equipments. Pertaining to the heavy duty engineering applications, the metal matrix composites have found their usefulness; hence engineers used it as an alternate to alloys. Most importantly the hybrid nanocomposites find their utility in engineering and technology. The behavior of individual elements in association with other constituent elements, provide a unique combination resulting in a novel material. The novel materials thus produced provide better resistance to wear, corrosion and toughness at reduced weight. Moreover this work has taken a hybrid nanocomposite reinforced in to a metal matrix. The Idea of this experimental work is to identify such a novel combination and analyze its machinability characteristics.

II. EXPERIMENTAL WORK

A. *Material selection*

Aluminium is selected as the base material. Since the objective is to develop hybrid reinforcement, it has been decided to choose a metal, a ceramic and an allotropic form of a carbon as the constituent elements. These Constituents are taken at different propositions. Table 1shown below gives the percentage composition of the elements.

TABLE I PERCENTAGE OF COMPOSITION OF SAMPLES

Composition no.	**% of Aluminium**	**% of Silicon Carbide**	**% of Magnesium**	**% of Graphite**
1	70	12	9	9
2	80	15	4	1
3	90	7.5	2	0.5

B. *Ball Milling*

The selected materials were subjected to mechanical alloying technique using a ball milling machine. The ball mill takes planetary motion for the size reduction process. The elements were individually milled for nearly 12 hours each and the hybrid composition was subjected to 8 hours of milling process. The details are furnished in the TABLE II given below. The speed set for the entire process in this "Skeletron" made ball milling machine was 350 rpm and at atmospheric ambience. The materials were kept in the air tight tungsten carbide coated vessel carrying 10carbide coated balls so as to maintain the powder(in grams) to ball (in numbers) ration of 10:1.

TABLE II MILLING TIME FOR INDIVIDUAL CONSTITUENTS

S.No	Material	Milling in Hours
1	Silicon Carbide	12
2	Magnesium	12
3	Graphite	12
4	SiC-Mg-Gr(for all the compositions)	8

C. Compaction and sintering

Green pellets were made using pneumatic compaction techniques. Dies were fabrication to make circular pellets of 40mm in diameter and 10mm in thickness. Poly vinyl Alcohol was applied as the binder to make pellets. A pneumatic pressure of 2.5 tons was applied to the fabrication of pellets. The compacted pellets were sintered in an induction furnace. The component was taken and kept in the alumina plate and placed inside the furnace for sintering and cooled inside the furnace. The furnace took 180 minutes to reach 550°c and the work piece was held for 30 minutes in open condition to liberate smoke particles. The details of sintering are given below in the TABLE III. After sintering, the pellets were tested for their hardness.

TABLE III SINTERING TEMPERATURE & TIME AFTER COMPACTION

Composition no.	Sintering Temperature in ^{0}c	Sintering Time in Hrs
1	350	1
2	550	3
3	550	3

C. Stir casting technique

Aluminium rods of about 700 grams were melted in a stir casting furnace at 1000^{0}C and then the Silicon Carbide-Magnesium-Graphite hybrid powder mixture in nano scale was pre-heated in a separate furnace at 500^{0}C.The pre-heated mixture was then mixed with the molten aluminium.The resultant composite was stirred at 300 rpm, maintaining the temperature at 1000^{0}C .The composite was then poured into the moulds, to take up the shapes of rods and plates. The rods of 20mm in diameter and 200mm in length and plates of size 10cm x 10cmx 1cm were cast.The compositions were maintained throughout the process.

D. Characterization

After the top-down nano synthesis under ball milling process the powder sizes and morphologies were verified using Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). The micro hardness of the pellets and cast materials were tested in Vicker's hardness testing machine. The tensile test was also conducted in the cast rod.

E. Machining using Wire-cut EDM

The plate like structure was chosen for machining under wire-cut EDM. The input parameters chosen for machining were Pulse duration, Pulse ON, Pulse OFF and servo feed. The performance measure were the Material removal rate (MRR), Surface roughness and Kerf Width. Taguchi's L9 Orthogonal array was chosen for the experimental design. The experiments were conducted and optimized using the mathematical concept Analysis of Variance (ANOVA).

III Results and Discussion

A. SEM Analysis

Figure 1 a-b shows the morphologies of as received matrix aluminium powder and the reinforcements as a mixture of Silicon Carbide, Magnesium and Graphite particles. The matrix material is relatively of a broad sized distribution with average size of 900 nm. The reinforcement particles are regular crystalline appearance with an average size of 2000 nm.

At the early stages of milling, it was found that reinforced particles are rarely seen within the matrix powder. The higher amount of reinforcement resulted in higher amount of particles embedded within the metal matrix. This behaviour may arise possibly due to hard reinforcement particles which may accelerate milling action since they act as milling media during mechanical alloying. The plastic deformation of aluminium the soft matrix starts immediately in short milling time leading to a change in its morphology from spherical to flattened shape. The processes of deformation at cold conditions and the subsequent welding can be observed from SEM images. It is apparent for the composite that the average size of particles increases as a result of welding of flattened particles.

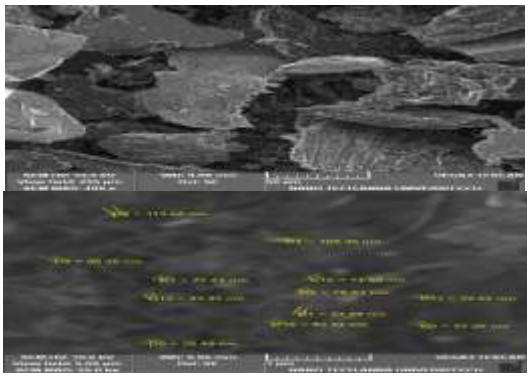

Figure 1(a) & (b) SEM images of particles after 6 hours of milling

From the Fig. 1 it is observed that the particles have an average size range of 80nm at 25kx magnification. The Nanoparticles appear to be randomly scattered. Since there was a time delay in obtaining the SEM results it is understood that the particles got agglomerated. The hard ceramic materials got disintegrated and easily reached the nano regime, while the ductile reinforcement copper and the lubricate substance graphite were relatively reluctant to breakdown to enter the regime of 10^{-9} scale.

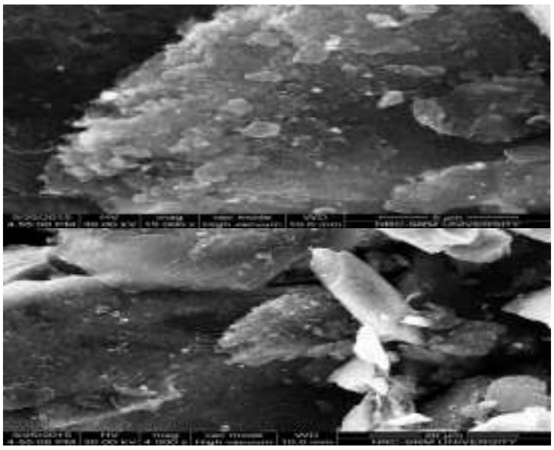

Figure.2 (a) & (b) SEM images of particles after milling for 12 hours

Increasing milling time, the composite powder containing coarse silicon carbide was heavily agglomerated into large particles. This means that composite powders with coarse reinforcement are more prone to assemble into a large one which suggests excessive cold welding. By subsequent milling for up to 16 hours causes slightly the reduction in particle size where fracture becomes dominant. It is noticed that reinforcement particles well dispersed inside the matrix after long time milling. Some large particles are still present inside the matrix even after 12 hours milling.

The milling process was smooth with the reduction in the percentage by weight composition of Graphite. The Images are clear with the reduction in the quantity of Graphite. Figures 2 (a) and (b) show the homogeneous distribution of particles in the sample taken. The reduction in graphite not only eased the milling process but also handy in the preparation of pellets in the cold pressing.

B. XRD

A Sample of was taken for XRD study to estimate the crystallite size of the powder, phase constituents, lattice strain. The crystallite size and lattice strain was calculated using the formula,

$$B\cos\theta = 0.9\lambda/d + \varepsilon \sin\theta \qquad (1)$$

Where λ is the wavelength of Cu Kα radiation in nm, d is the crystallite size in nm, θ is the Bragg angle, ε is the lattice strain and B is the Full width half maximum of the ray in nm. The XRD tests were conducted individually to all the elements to ascertain their entry into the nano regime. The XRD pattern for SiC is shown in the figure 3 as a sample.

TABLE V DETAILS OF XRD RESULTS OF NANOCOMPOSITE POWDER

D	2Theta	I (rel)	I (abs)	FWHM
3.3575	26.5265	18	21	.2425
2.6040	34.4131	42	48	.2000
2.4500	36.6500	51	74	.2320
D	2Theta	I (rel)	I (abs)	FWHM

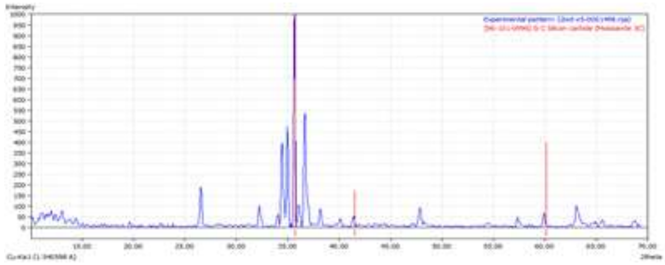

Figure 3. The XRD pattern for the Silicon Carbide powder after reducing it in to nanao scale.

From the output results obtained from the X-Ray Diffraction studies as shown in the TABLE V, with the help of the data base provided by International council for Powder diffraction studies (ICPDS) and supported by Material report Analyzer software, we compared the peak values of all the constituent elements with the values available in the database and confirmed the presence of all the constituent elements.

With the same results it is possible to find the morphology and the lattice crystal structure of the constituent elements at the nano scale. Substituting the values for the parameters in the Scherrer's formula the particle size reaching the nano regime has been confirmed.

C. Hardness

TABLE VI HARDNESS FOR PELLETS OF VARYING COMPOSITION

Composition	Compacting Pressure	Sintering temperature &time	Vickers Hardness
70% Al, 30% Hybrid	250 bar	350^0C 1 hr	53.77 VHN
80% Al ,20% Hybrid	250 bar	550^0C 3 hr	53.78 VHN

TABLE VI and VII gives hardness values of compaction and stir cast products. The Hardness of the stir cast product is better than the compacted and the sintered products. The composition shown in the table 7, and its subsequent stir cast showed the positive sign during the hardness and tensile tests, to take up machinability analysis

TABLE VII HARDNESS FOR STIR CAST PRODUCT.

Composition No	Rockwell Hardness	Vickers
70%Al,30%(Hybrid)	81HRC	90.5 HV

TABLE VI, shows the details of hardness values measured in Vicker's Hardness measuring instruments. The hardness values kept increasing with the increase in the base metal. It is obvious that with the increase in reinforcements known for their high hardness, the hardness of the compacted pellets should increase. The results show the adverse effect of the hybrid inclusion. The reason for the unexpected change in hardness is that the constituent elements should find a bonding among themselves upon application of force and at the time of sintering. Due to the inadequacy in pressure, load applied in making the pellets and the combined effects of sintering temperature and time, only the homogeneous substance got the reliable bonding, that is the pure aluminium had its bonding together, where as the other ingredients couldn't find the needed cohesion.

The consequential reasoning for the increase in hardness with increase in base metal is certainly the inadequacy in compacting force and sintering process. The pellets thus formed could not be used for machining process. Though they showed good hardness they crumble at the application of external load. Whereas the needed hardness is achieved in the plates and rods cast using the stir casting technique.

D. Tensile test

The tensile test is conducted to check the tensile strength of the developed composite material. The cast rod is cut into half the size from its original dimension that is from 200mm length to 100mm length. The test specimen is prepared with standard dimensions required for tensile testing in Ultimate Tensile Testing.

TABLE VIII RESULTS OF THE MECHANICAL PROPERTIES

Material	Aluminium	Hybrid composite
Brinell hardness(BHN)	30-33	55
Yield strength(kN/mm^2)	52	67
Ultimate tensile strength (mPa)	110-124	109

The ultimate strength of the aluminium is found to be 124 mPa. This value is compared with the ultimate strength of our composite material. The report attached as shown in the Figure 5 is for the tensile test of the developed material. The graph shows the stress vs strain curve and it is clearly given that the Ultimate strength of the composite material is 0.156 KN/mm^2 that is 156MPA which is higher than the strength of aluminium 6061 grade. The Aluminium used for melting in stir casting process is 6061, the new composite material shows an increase in the Ultimate strength by 20.5%. Thus a better mechanical property is achieved in the new hybrid metal matrix composite.

(a)

(b)

(c)

Figure 4 (a),(b),(c) Represents the images of rod used for tensile test

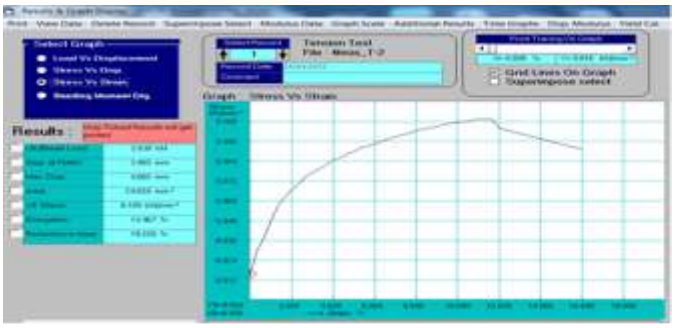

Figure 5. The Stress-Strain Curve obtained from the tensile test on the nano hybrid reinforced composite

E. WIRE EDM Machining

The obtained plate from stir casting was machined using Wire EDM machine by giving suitable input parameters, the figure 6, shows the plate before and after machining wire EDM process

Fig 6 Cast plate before and after Wire EDM

From the input and output values of the Wire EDM process the needed optimizing parameters kerf width, surface roughness, material removal rate are calculated the surface roughness value is taken from the surf coder instrument and the material removal rate is calculated using the MRR formula, fig below shows the calculated value for optimizing

TABLE XI EXPERIMENTAL RESULTS & CALCULATED KERF WIDTH, SURFACE ROUGHNESS AND MRR

Experiment .No	T_{on}	T_{off}	Wire feed	Gap volt	Current	M/c Speed	Time	Kerf width	Ra	MRR
1	4	8	5	55	1.5	4	3.43.63	0.378	3.23	19.80
2	4	6	6	60	1.2	3.4	4.12.15	0.355	3.25	15.50
3	4	9	7	65	1	3.1	4.41.41	0.377	3.2	15.37
4	3	6	6	65	1.2	3.2	4.26.43	0.384	3.9	16.20
5	3	8	7	55	1.5	3.4	4.12.45	0.352	3.16	15.36
6	3	9	5	60	1.2	3.1	4.35.23	0.352	3.18	14.55
7	5	6	7	60	1.7	3.8	4.51.86	0.398	3.25	17.82
8	5	8	5	65	1.1	2.8	5.01.71	0.374	3.13	13.41
9	5	9	6	55	1.7	3.8	3.51.45	0.381	3.24	19.51

F. *Optimization using Taguchi's Method*

The Taguchi method has been adopted to analyse the experimental results. Kerfwidth, Surface roughness and Material removal rate are considered as the output parameters. The signal to noise ratio is taken as the pivotal aspect to compare against the input parameters Pulse On time, Pulse OFF time,wire Feedand Gap Volatge.

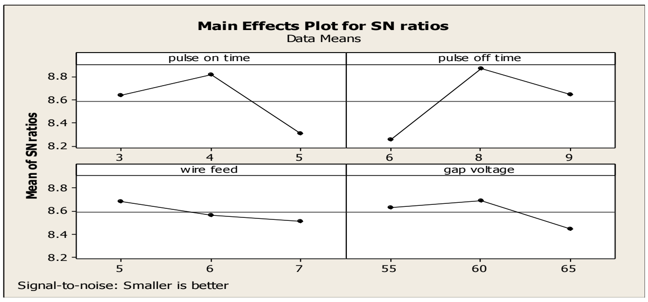

Figure 7 Main effects kerf Width and the Plot for SN ratios

Kerf width is one of the important performance measures in WEDM. Kerf width is the measure of the amount of the material that is wasted during machining. It determines the dimensional accuracy of the finishing part. It is understood that smaller is the better. The input parameter Gap Voltage alone gives that higher the gap voltage higher is the deviation from the mean SN ratio that is lower the SN ratio. It is predictable from the kerf width point of view Gap voltage would be a vital parameter in reaching the desirable output in this Wire-cut discharge machining. The Regression equation is given as: "Kerf Width = 0.35169 - 0.0015 pulse ON time - 0.00378571 pulses OFF time - 0.004 wire Feed + 0.0012 Gap Voltage". In order to have a better kerf width the Pulse ON time has to be high, the wire feed has to be between low and high to have a better kerf width. Similarly as explained earlier the gap voltage should be at an intermediate value of the three input value gap voltage to have a lower kerf width. Apart from the experiment 1 all other values follow the line depicting the normal distribution. The reason for the deviation in the first experimental value is the error in calculating time or in observing the gap voltage.

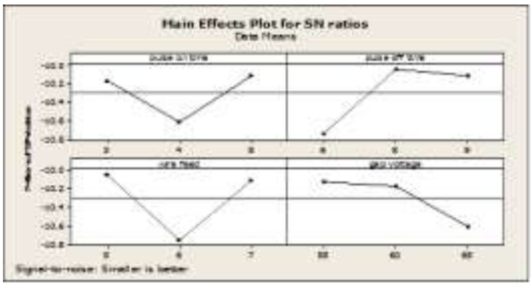

Figure 8 Effects of Surface roughness and the plot for SN ratios

Roughness plays an important role in determining how a real object will interact with its environment. Rough surfaces usually wear more quickly and have higher friction coefficients than smooth surfaces. Least Surface roughness value gives the good surface finish. There are many number of evaluation parameters for measuring the surface roughness values among that RA parameter is selected as per the surface tester. Similarly from the diagram it is inferred that smaller the SN ratio it is better for all the 4 prominent input parameters. "Ra = 0.967 + 0.0217 pulse ON time + 0.0643 pulse OFF time + 0.115 wire Feed + 0.0193 Gap Voltage". The surface roughness is better at lower level of the input parameters Pulse ON, Pulse OFF and Gap Voltage. The input parameter wire feed alone shows the slight variation that medium level of wire feed improves the surface finish. Apart from the experiment 2 all other values follow the line depicting the normal distribution. The reason for the deviation in the first experimental value is the error in calculating time or in observing the values of parameters.

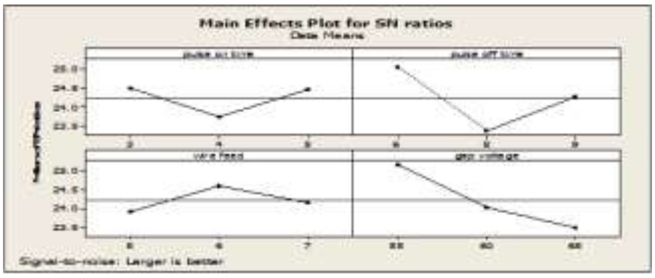

Figure 9 Effects of MRR and the plot for SN ratios

The Material Removal Rate (MRR) in WEDM operations is The volume of material/metal removed per unit time in mm^3/min. For each wire cutting of the work piece, some scraps of material are removed. Experimental data for the MRR have been reported in graph. Material Removal Rate is being a **'Higher the better'** type of machining quality characteristic, therefore the effect of process parameters on material removal rate is depicted with the help of the graph. From the graph it is observed from the graph that Pulse ON and Wire feed should be at high level, whereas the Pulse OFF time and Gap voltage should be low. "MRR = 30.1 + 1.24 pulse ON time - 0.580 pulse OFF time + 0.145 wire Feed - 0.207 Gap Voltage". Apart from the experiment 2 all other values follow the line depicting the normal distribution. The reason for the deviation in the first experimental value is the error in calculating time or in observing the gap voltage

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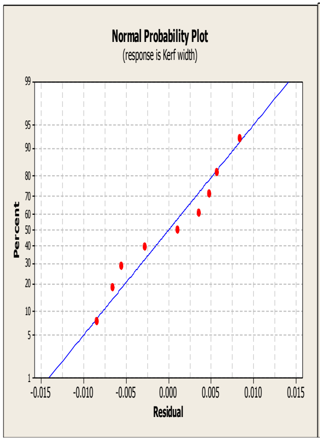
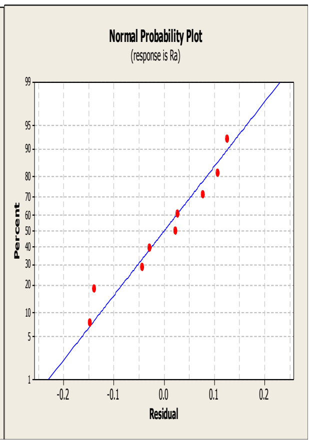
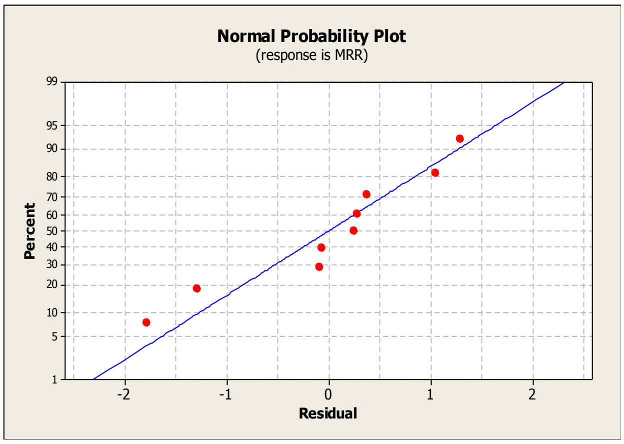


(a)



(b)



(c)

Figure 10 (a),(b) and (c)., Normal Probability Plot for Kerf width, Surface roughness and MRR respecttively

3.*7 ANALYSIS OF VARIANCE* (ANOVA)

The basic idea behind ANOVA is to break down the total variability (of the experimental results) into components of variance and then access their significance. The variation components will be those associated with factor effects (main or interaction effects) and that associated with random variation.

TABLE 10 ANALYSIS OF VARIANCE TABLE

ANOVA						
Source	DF	SEG SS	ADJ SS	ADJ MS	F	P
Pulse ON Time	1	0.0003082	0.0003082	0.0003082	1.30596	0.316882
Pulse OFF Time	1	0.0006178	0.0006178	0.0006178	**2.61807**	0.180963
Wire Feed	1	0.0000882	0.0000882	0.0000882	0.37363	0.574061
Gap Voltage	1	0.0000960	0.0000960	0.0000960	0.40683	
Error	4	0.0009439	0.0009439	0.0002360		
Total	8	0.0020540				

From the Table X**,** it is observed that the variance in a particular input parameter plays a significant role in determining the quality of output in terms of surface finish and Material removal rate. The maximum variance value **F=2.61807** against the Pulse OFF Time indicates that Pulse OFF Time has a telling effect on the optimum combination of input parametric values to effect an optimally machined material.

CONCLUSION

A unique combination of two metals, a ceramic and an allotropic form of carbon was tried in this experimental work. Several proportions of the aforesaid combinations at Nano scale were tried**.**

- In this work, the nano -composite powder of Aluminium, Silicon Carbide, Aluminium and graphite was synthesized by mechanical alloying technique, ball milling process. The solid – state reduction reaction in powders milled for 6 hours is attributed to an increase in the reactivity of the ingredient powders arising from the modification of crystallite size lattice strain as well as lowering the particle size of both phases.
- The reactive nanocomposite powder was compacted into pellet of 40 mm diameter and 10 mm thick by cold isostatic pressing process under the load of 2.5 tons. The pellet was sintered in atmospheric conditions at 550⁰C for duration of 3 hours. The mechanical properties like mass Vickers hardness was measured under green and sintered condition. The results were not so appreciable because of the presence of graphite and its implications in the bonding. It was lead to the reduction of graphite's composition in % by weight. The percentage by weight composition of all the constituent elements is changed and ball milled for 12 hours individually and for 8 hours with the mixture.
- The phase constituents of end product was found by XRD test and confirmed the presence of all the ingredients, their morphologies and the new intermetallic phases. After long duration of milling, a homogeneous distribution of reinforcements in the composite powder with smaller particle size was achieved. The particles show an average size of 80nm in cross section when analyzed through Scanning Electron Microscope (SEM).
- The pellets were made with a compaction load of 2.5 tons, and sintered at 550°C for a time duration of 3 hours. The Vickers hardness of pellets was measured, the values are 53.78 HV. Plate and Rod like structures were made with suitable dimensions using stir casting technique. The hardness values measured for the structures are 68.40 HV
- The tensile strength of the material is measured as 109 N/mm^2 . The Micro structural analysis of the plate was

done and confirmed the presence of constituent elements in random locations.

- The Plate developed using the metal matrix composite was machined using **Wire-cut EDM** after designing the experiments for a lay out of 9 combinations **(L9**), with Pulse ON, Pulse OFF, Wire feed and Gap Voltage were chosen as input parameters.
- The experimental results were analyzed for single objective optimization using Taguchi method. The software **Minitab** was made use of. It has been observed that Pulse OFF Time is the input parameter among the other input parameters that plays a vital role in the optimized results. It has been concluded that the materials chosen can be reduced to Nanoscale and still be cast in to a plate or a rod with good mechanical properties. The same material can be machined with optimized input parameters in a Wirecut Electrical Discharge Machine.

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