STUDY OF MECHANICAL AND WEAR PROPERTIES OF HYBRID COMPOSITE MATERIALS WITH NANO FILLERS FOR ELEVATED TEMPERATURE APPLICATIONS

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ABSTRACT *Hybrid composites by varying glass fibre reinforcement and fillers will result in a composite material which has enhanced thermal properties and can withstand at elevated temperature in the range of 2000C. The fillers usedshows thermal properties like high service temperature, low coefficient of thermal expansion, maximum operating temperature, high melting point, retention of mechanical properties at elevated temperatures etc. Polymer composites, in current scenario can withstand temperatures up to 1400C-1800C.These composites, shows increased mechanical properties at high temperature due to presence of Multi-Walled Carbon Nanotubes. Further the increasing cost of metals i.e. Multi-Walled Carbon Nanotubes gave way for research of high temperature application polymer composites.*

Keywords: *Elevated Temperature resistance, Activated Carbon, Chopped E-Glass, Al2O3, SiC, BaSO4, Multi-Walled Carbon Nanotubes.*

1. INTRODUCTION

A composite material can be defined as a combination of two or more materials that results in better properties than those of the individual components used alone. In contrast to metallic alloys, each material retains its separate chemical, physical, and mechanical properties. The two constituents are reinforcement and a matrix. The main advantages of composite materials are their high strength and stiffness, combined with low density, when compared with bulk materials, allowing for a weight reduction in the finished part.

Continuous-fiber composites are often made into laminates by stacking single sheets of continuous fibers in differentorientations to obtain the desired strength and stiffness properties with fiber volumes as high as 60 to 70 percent.Fibers produce high-strength composites because of their small diameter; they contain far fewer defects (normallysurface defects) compared to the material produced in bulk. As a general rule, the smaller the diameter of the fiber, thehigher its strength, but often the cost increases as the diameter becomes smaller.

The most common advanced composites are Polymer Matrix Composites (PMC's).A polymer is a large molecule, or macromolecule, composed of many repeated subunits, known as monomers. Because of their broad range of properties, both synthetic and natural polymers play an essential and ubiquitous role in everyday life. While the polymers are used extensively in everyday life, their industrial applications are very few due to their low strength, low melting point and other properties.

These properties, however, can be enhanced by reinforcing the polymer with materials with high strength and melting point and by adding fillers which also help in stability of the material. The polymer matrix, together with known quantity of reinforcing fibers and the fillers, is called a Polymer composite. The reinforcing fibers used in polymer composites are usually made of either carbon or glass. These fibers are present either in crystalline form or amorphous form. The fillers are organic or inorganic compounds which usually rigid and immiscible with the matrix in both molten and solid states, and, as such, form distinct dispersed morphologies. The polymer matrix material or binder is usually a thermoset. Commonly used binders are Epoxy resins, Polyamide and Polyimide resins.

Recently, research has been extended to evaluate the effect of nanoparticles on mechanical properties of polymer composites. One of the promising nanoreinforcements was carbon Nanotubes (CNTs) which has exceptionally high mechanical properties improvement of polymer composite as a result of CNTs addition was reported in few studies.

2. LITERATURE SURVEY

Karen I. Winey et.al.,[1]Observed that the effects by the addition of carbon nano tubes During flammability experiments, nanotube/polymer composites with well -dispersed nanotubes form a free standing nanotube network that remains robust after burning, thus serving as a protective layer and providing good flame – retardant effectiveness, Carbon nanotubes provide a new regime of fillers because of their high electrical conductivities, large aspect ratios, and nanometer-scale diameters and have revitalized the investigation of percolation phenomena.

Nanda Gopal Sahooet.al., [2] Experimented theCNT fictionalization and matrix polymer design for dispersionof CNTs and interfacial adhesion between CNTs anda polymer matrix are the key challenges for development of high performance CNT Composites.A specific functionalization of CNTs is required for strong interfacial adhesion between CNTs and a given polymer matrix, which may also simultaneously improve the dispersion of CNTs in the polymer matrix.

Md Nadeem et.al., [3]Observed that

I. The impact energy for the composite having 5% SiC and 15% Al2O3 has shown the maximum impact strength, which is due to the fracture toughness of both SiC and Al2O3 are maximum in comparison with other fillers.
II. 10% Al2O3 and 10% SiC has shown minimum specific wear rate and could be considered as optimum percentage of these fillers.
III. The tensile strength increased is 63.4% which is very significant this is due to high tensile modulus of both SiC and Al2O3.
IV. The general conclusion is that the addition of SiC and Al2O3 has significantly contributed to the improvement in wear resistance, impact and tensile strength predominantly by Al2O3.

3. OBJECTIVES

- To identify suitable polymer matrix, reinforcements and filler materials based on the available literature.
- To fabricate the required composite as per ASTM standards.
- To develop a hybrid composite material.
- To evaluate the mechanical properties of a developed composite.
- To conduct tests on hardness, tensile strength & impact strength at elevated temperatures.
- To analyze and to validate the results of the tests using Scanning Electron Microscope (SEM) micrographs.
- To analyze and validate the experimental results.

4. METHODOLOGY AND EXPERIMENTAL SETUP

The methodology adopted for this investigative work is delineated below

1. Procurement of raw materials.
2. Calculation of proportions.
3. Fabrication of epoxy based hybrid composites.
4. Conduction of tests.

Five different compositions of composites were made by blending all seven components and varying the percentage of $BaSO_4$, Activated Carbon Powder and MWCNTs. The specimens were fabricated at Sri Jayachamarajendra collage of engineering, Mysore. The content of each composition of composites is given in Table 1.

Table 1: Composition of Specimen in Weight.

Specimen No / Components By Weight	1 (gms)	2 (gms)	3 (gms)	4 (gms)	5 (gms)
Epoxy Resin	120	120	115	110	110
Aluminium Oxide	69.52	69.52	69.52	69.52	69.52
Silicon Carbide	56.32	56.32	56.32	56.32	56.32
Chopped E-Glass Fiber	23.06	23.06	23.06	23.06	23.06
E-Glass powder	14.52	14.52	14.52	14.52	14.52
Activated Carbon Powder	11.46	25.87	40.29	54.70	69.12
Barium Sulphate	126.72	95.04	63.36	31.68	0
Multi-Walled Carbon Nanotubes	0.48	0.53	0.58	0.63	0.69
Total (gms)	422.08	404.86	382.65	360.43	343.23

Specification and fabrication of Mould plate:

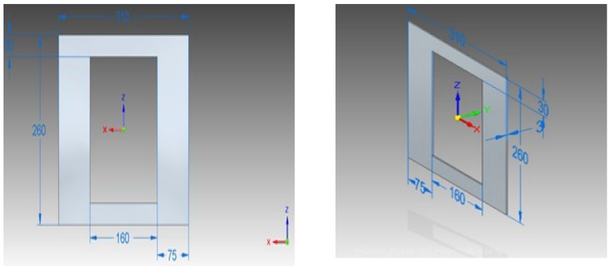

Figure: 1 Design specification of mould plate

Figure: 2 Weighing and mixing of all powdered components

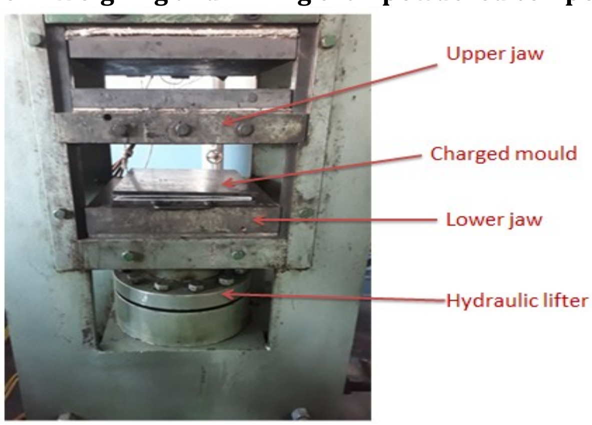

Figure: 3 Hydraulic press

First the materials are weighed as required. Then they are put together and mixed well. The mixture is then poured into a pre-prepared mould of the required thickness is shown in figure 1. It should be noted that the Epoxy resin and Hardener start to set i.e. start solidifying after 30 minutes of mixing and hence, the mixture should be poured into the mould before the setting time is shown in figure 2. The mixture is poured in excess and compressed in hydraulic press. The whole process of pressing is shown in figure 3 Similarly, five different compositions are poured in separate moulds by varying the Nano silica powder and MWCNTs content while keeping all the other weight percentages constant. The content of Activated Carbon Powder, $BaSO_4$ and MWCNTs are varied in steps of percent in such a way that in any composition, the sum of Activated Carbon Powder, $BaSO_4$ and MWCNTs is 20 percent of the total weight. The moulds are then left for 24 hours to solidify and cure at room temperature. After solidification, the specimens are removed from the mould and post cured at 100^{o}C for 12 hours in a hot air oven. The completed plate is as shown in the figure

4. The specimens are then taken out and labeled. The specimens are then marked as per the test standards. Specimens were prepared for tensile, impact strength and hardness test.

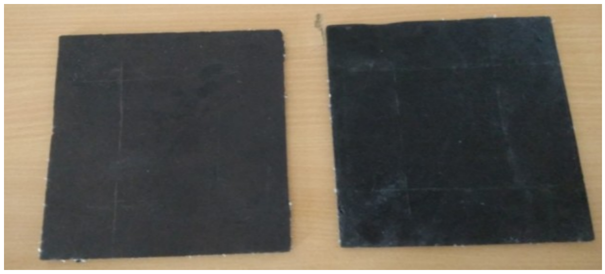

Figure 4: Fabricated composite plate

Testing for mechanical properties of Epoxy Based Hybrid Composites

In order to investigate the mechanical properties tests like tensile, impact and hardness tests are conducted. Details of the experimentation and standards used for them are discussed below.

4.1 Tensile Test:

Tensile tests were conducted as per the ASTM D-638. Computerized Universal Testing Machine (UTM) was used for this purpose and the loading arrangement is shown in Figure. 4.1(a) Specifications are also mentioned. The dimension of the tensile specimen was 165 mm x 19 mm x 3.2 mm. Gauge length was 50 mm. Results were used to calculate the tensile strength composite samples.

Figure: 4.1(a) - Tensile testing machine setup (UTM).

Specification of UTM:

Make: International equipment, India
Model: PST-VGST/KFIST-05
Capacity: 50KN
Jaw separation speed: 1 to 500mm/min

The specimen used for Tensile test is shown in the figure 4.4.1 (b) which was prepared according to ASTM standards.

Figure: 4.1(b) – Tensile specimen.

4.2 Impact Test:

Izod impact tests were conducted on V-notched composite specimen according to ASTM D256. A Pendulum impact tester, shown in Figure 4.2(a) was used for this purpose. Dimension of the specimen were

64 mm x 12.5 mm x 3.2 mm. The pendulum impact testing machine ascertains the impact strength of the material by shattering the specimen with a pendulum hammer, measuring the spent energy and relating it to the cross section of the specimen. The respective values of impact energy of different specimen are recorded directly from the digital indicator and reported.

Figure: 4.2(a)-Computerized impact testing machine

Specification of impact testing machine:

Capacity: Up to 25.00 Joules.
Release angle of pendulum: 150 degree.
Range of four scales: 0-2.71 Joules, 0-5.42 Joules,
0-10.84 Joules, 0-21.68 Joules and 0-25.00 Joules.
Power: 230 Volts, 50Hz, single phase.

The specimen used for impact test is shown in the figure 4.2 (b) which was prepared according to ASTM standards.

Figure: 4.2(b) – Impact Specimen.

4.3 Hardness Test:

Shore-D hardness tests shown in figure 4.3 were conducted on specimen according to ASTM D2240 using Durometer. The hardness tester is placed on the specimen and pressure is applied so that the flats underneath the tester touch the surface of the specimen. The readings are taken directly from the dial. The specimens are then heated to different temperatures and the readings are taken to determine the variation in the hardness of the specimen with respect to temperature.

Figure: 4.3 - Shore-D hardness testing machine.

Specification of hardness testing machine

Make: Hiroshima Company
Range: 0 to 100

4.4 WEAR TEST:

Wear is defined as the action of causing deterioration thought use the molecular science wear is considered to be the erosion of material from a solid surface by the action of another solid.

Friction: friction is the force that resists relative motion between two bodies in contact. **Coefficient of friction**: the coefficient of friction (COF) often symbolized by the greek letter μ is a dimensionless scalar value which describes the ratio of the force of friction between two bodies and the force pressing them together.

Testing methods:

Wear test was carried out using Taguchi's Method using L-16 array as shown in the table 2

Table 2: Taguchi's Method using L-16 array

	Composition	Load (Kgs)	Speed (m/s)	Sliding Distance (m)
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	1	4	4	4
5	2	1	2	3
6	2	2	1	4
7	2	3	4	1
8	2	4	3	2
9	3	1	3	4
10	3	2	4	3
11	3	3	1	2
12	3	4	2	1
13	4	1	4	2
14	4	2	3	1
15	4	3	2	4
16	4	4	1	3

Specification of wear test specimen and test pin are shown in the table 3, based on which specimens were prepared and tested.

Table 3: Wear test specification

Sl No	Tests	Standards	Specimen dimension in mm (length*breadth*thickness)
1	Wear	ASTM G-99-05	(10×10×3.5)

Wear test specimen is now pasted on the pin and the testing is carried out. The specimen pasted on the pin is shown in the figure 4.4 (a).

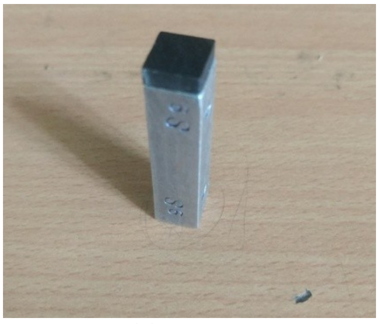

Figure 4.4(a) - Wear Specimen

PIN ON DISC:

Fig. 4.4 (b) - Pin On Disc Machine

Working:

Pin on disc type machine is shown in figure 4.4(b) designed to study the friction and wear properties in sliding contacts. Siding occurs between a stationary pin/ball and a rotating disc at affixed rpm/speed. the disc is driven by a 2067 HP motor capable of running at 0-2950 rpm .The stationary upper specimen pin holding the ball of the required diameter can be loaded manually (or automated by the software optionally). Precise loading can be done manually or through software (in automated version). piezo electric sensor is used for the measurement of frictional force.

Specifications of pin on disc:

Table 4.5 Pin on Disc machine specification

Parameter	Units
1.Pin size	10 mm
2.Disc size	50 to 140 mm
3.Disc rotation speed	1-2950
4. Wear track diameter. mean	50-100 mm
5.Load	5-200N (any steps possible)
6.sliding speed Range	0-10 m/s
7.power	230V, 50 Hz S phase

Test Procedure:

The testing procedure has been conducted for four different operating conditions:

1. By varying the sliding distance.
2. By varying the load.
3. By varying the speed.
4. By varying the composition.

Calculation:

Formula used:

Sliding distance (SD) = (π×D×N×T)/1000 in meters.

Where D-diameter (two times the track radius, in mm)

N-RPM of rotating disc

T - time in seconds

Specimen calculation:

Composition: 9.3% of NSP + 0.7% MWCNT

Track radius R= 40mm

RPM = 477.46

Sliding distance = 500m

To calculate 't"

500 = (π×80×477.46×t)/1000

T = 4.16 = 4min 16sec.

Hence it can be concluded that if the machine runs for 4min 16sec with a specimen track radius of 40 mm and speed 477.46 RPM, it will cover a sliding distance of 500 m.

5. RESULTS AND ANALYSIS

Physical and mechanical properties describe the behavior of materials when they are used in practical applications. The properties such as hardness describe the physical state of the system. The mechanical property of the material is a measure of the behavior of the material under different loading conditions. Tests were done to notice the effect of variation of filler content on the physical and mechanical properties.

5.1 Effect of $BaSO_4$, Activated Carbon Powder and MWCNTs on tensile strength of Epoxy Based Hybrid Composite Material

The tensile strength is an engineering value that is calculated by dividing the maximum load on the material by the initial cross sectional area of the test specimen. The Table 5.1 shows the results obtained during the tensile test conducted on all the five specimens at elevated temperature.

Table 5.1 – Tensile Strength of Epoxy based hybrid composites.

Specimen No.	Filler Content	Impact Energy (J)			
		32° C	50° C	100° C	150° C
4	15.2% ACP,4% $BaSO_4$,0.8% MWCNTs	1.6	2.4	2.2	0.9
5	19.1% ACP,0% $BaSO_4$,0.9% MWCNTs	4.5	3.9	2.20	0.7

The graph for the tensile strength is shown in Figure 5.1.

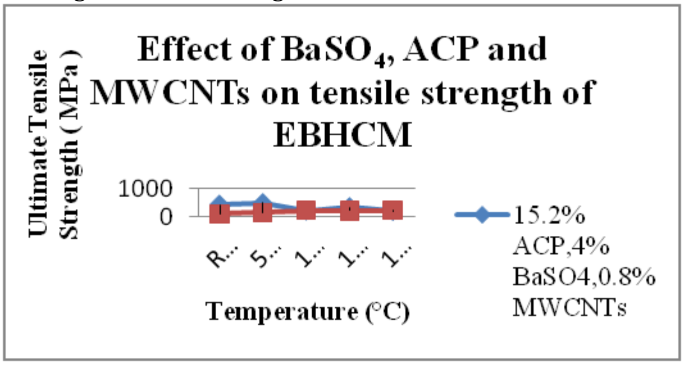

Fig.5.1 – Effect of $BaSO_4$, ACP and MWCNTs on tensile strength on EBH Composites

From the graph it can be seen that tensile strength is highest in the composite with 3.5% ACP,16% $BaSO_4$,0.5% MWCNTs at 120°C and lowest in that with 19.1% ACP,0% $BaSO_4$,0.9% MWCNTs at 120°C. Since the $BaSO_4$ has high tensile modulus and can withstand more loads and at the same time and can transfer load to adjacent particles as well, It also reduces the load concentration at points which in turn reduce the stress concentration, thereby increase the modulus of specimen. Hence by increasing the$BaSO_4$, the Epoxy Based Hybrid Composite Material's ultimate tensile strength increases at 120°C. Also at 150°C we can observe that the ultimate tensile strength of the Epoxy Based Hybrid Composite Material decreases with the decrease of $BaSO_4$. But at a certain point, when there is 0% of$BaSO_4$ present in the composite, the ultimate strength increases drastically due to presence of high amount of MWCNTs and this phenomenon is called Synergic effect.

5.2 Effect of $BaSO_4$, Activated Carbon Powder and MWCNTs on impact strength of Epoxy Based Hybrid Composite Material

The material's resistance to fracture is known as toughness. It is the energy absorbed by the material before fracture and is expressed in terms of the same. A ductile material can absorb considerable amount of energy before fracture while a brittle material absorbs very little energy before fracture. Table 5.2 shows the results obtained during the impact test conducted on all the five specimens.

Table 5.2 – Impact strength of CGRP Composites.

Specimen No.	Filler Content	Impact Energy (J)			
		32° C	50° C	100° C	150° C
4	5.2% ACP,4% $BaSO_4$,0.8% MWCNTs	1.6	2.4	2.2	0.9
5	9.1% ACP,0% BaSO4,0.9% MWCNTs	4.5	3.9	2.20	0.7

The graph for the impact strength is shown in Figure 5.2

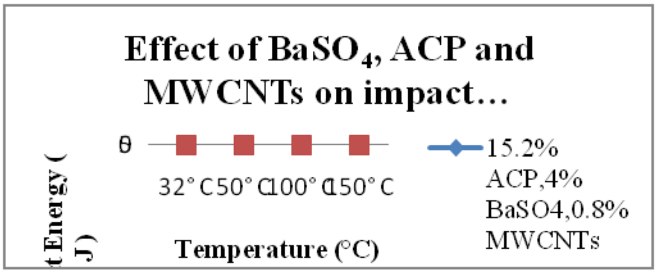

Fig. 5.2 - Effect of $BaSO_4$, ACP and MWCNTs impact strength on EBH Composites

From the table 5.2 it can be observed that the impact energy is highest for the composite having 15.2% ACP,4% $BaSO_4$,0.8% MWCNTs and is lowest for the composite having 3.5% ACP,16% $BaSO_4$,0.5% MWCNTs. From the same table it can also be seen that the increment of activated carbon powder and multi walled carbon nano-tubes, the fracture toughness rises correspondingly. It could also be seen that (from the table 5.2 and Fig 5.2) the Impact strength is increased from the EBH composite with 3.5% ACP,16%

$BaSO_4$,0.5% MWCNTs to EBH composite with 15.2% ACP,4% $BaSO_4$,0.8% MWCNTs by 63% and further the impact has reduced by 30%.This clearly shows that addition of ACP and MWCNTs increases the impact strength of designed composite significantly, this is due to the fact that breaking of ACP and MWCNTs particles require more impact load because the fracture toughness of both the ACP and MWCNTs are high.

5.3 Effect of $BaSO_4$, Activated Carbon Powder and MWCNTs on hardness of Epoxy Based Hybrid Composite Material

Hardness is a measure of how resistant a solid matter is to various kinds of permanent shape change when a force is applied. The hardness readings of the specimens were obtained directly from the Shore-D hardness tester. The specimens were heated to six different temperatures by a hot air oven and the readings were taken. Table 5.3 shows the readings thus obtained

Table 5.3 – Effect of temperature on hardness of EBH Composites

Specimen No.	Filler Content	Temperature (°C)				
		32°C	50°C	100°C	150°C	180°C
		Shore-D Hardness no.				
4	15.2% ACP,4% $BaSO_4$,0.8% MWCNTs	90	75	77	81	70
5	19.1% ACP,0% $BaSO_4$,0.9% MWCNTs	88	69	64	72	70

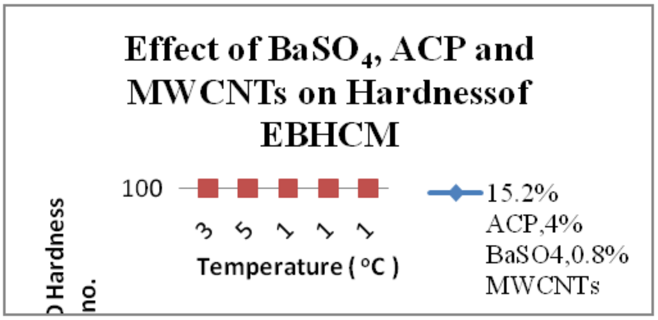

Fig. 5.3 – Variation of Shore-D hardness of EBH Composites with elevated temperature

The above table 5.3 and Fig 5.3 shows that, at elevated temperatures the composite with 19.1% ACP,0% $BaSO_4$,0.9% MWCNTs has the highest hardness and the composite with 3.5% ACP,16% $BaSO_4$,0.5% MWCNTs has the least hardness. This is because of presence ofhigh amount of both ACP and MWCNT. The interference is that higher percentage of both ACP and MWCNT contributes to retention of hardness since thermal conductivity of ACP and MWCNT are very much higher. This makes ACP and MWCNT to conduct away the heat more rapidly thereby retaining its hardness. Thus we can say that the depletion of the amount of $BaSO_4$ does not have any effect on the hardness of the composite.

It can be seen that the Shore-D hardness number of all the above composites decreases slightly with an increase in the temperature (32°C, 100°C,125°C&150°C). But while looking down the table, by increasing the percentage of both ACP and MWCNTs in all the composites, the Shore-D hardness number also increases at different temperatures(32°C,100°C,125°C &150°C) as shown in table 5.3.

5.4 Effect of ACP and MWCNTs on Wear properties of Epoxy Based Hybrid Composite Material.

Table 5.4: Process parameters

Factors	1	2	3	4
Composition(gms)	0	0.48	0.63	0.69
Load(Kgs)	1	2	3	4
Speed(m/s)	2	3	4	5
Sliding Distance(m)	500	1000	1500	2000

Minitab 16 Software was used to analyze S/N ratio of parameters used to build models in Compression Moulding. Typical monitored response plots on wear test based on S/N ratio are shown in figure 5.4.1.

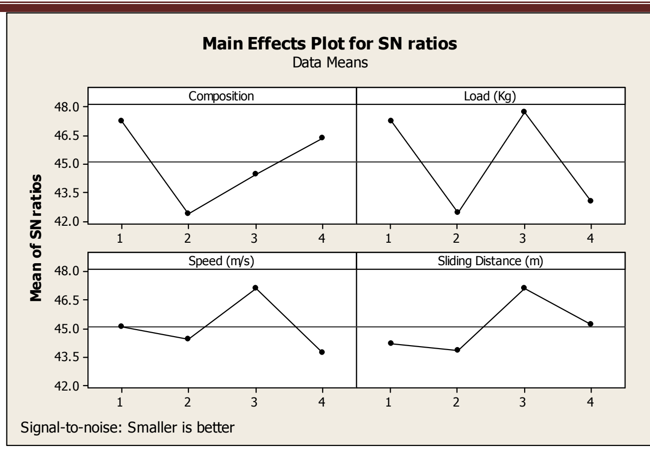

Figure 5.4.1: Main effects plot for wear test

From the figure the main effect plots for composite material is that, the factor speed has lowest effect on wear loss as the response variable. The optimum level for a factor is the level that gives lowest values in the experimental region.

From the table (5.4) and figure (5.4.1) it is clear that

a. With zero percentage of multi walled carbon Nano tubes, the material has taken maximum wear loss while composition with higher percentage of MWCNT,s have taken least wear loss
b. Wear loss is maximum for the material with zero percentage of MWCNT's while the material retains its behavior at higher loads and has taken least wear loss.
c. It can be observed from the graph that the material has given excellent wear resistance with higher percentage of MWCNT and higher speeds. Hence the material has excellent wear resistance at higher speeds.
d. From the graph it is clear that the wear loss is maximum as the sliding distance increases. Hence the material has given least resistance at higher sliding distances.

Conclusion

1. The impact energy for the composite having 14.8% ACP and 1.2 % MWCNTs has shown the maximum impact strength at room temperature and the plate with 14.8% ACP and 1.2 % MWCNTs has given highest impact strength at 32^{0}C, which is due to the fracture toughness of both NSP and MWCNTs are maximum in composition with other fillers.
2. The tensile strength decreased with increase in temperature **which** is very significant. This is due to high tensile modulus of MWCNT at 180°C. But the ultimate strength increases drastically in the EBH composite 18.7% ACP and 1.3% MWCNTs (at 180°C) due to higher concentration of MWCNTs and this phenomenon is called Synergic effect.
3. At elevated temperatures, higher percentage of ACP contributes to retention of hardness. since thermal conductivity of ACP and MWCNT are very much higher, this makes NSP and MWCNT to conduct away the heat more rapidly thereby retaining its hardness.
4. At higher loads 18.7% ACP and 1.2% MWCNTs shows a better behavior for wear properties.

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