Studies on Milling Operations on Graphene Oxide-Epoxy Nano composites: Optimization by Taguchi Approach

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Submitted By:

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CANDIDATE'S DECLARATION

I Hitesh Goel, 2K15/PTE/12 student of M.Tech Polymer Technology hereby declare that the project dissertation titled "**STUDIES ON MILLING OPERATIONS ON GRAPHENE OXIDE-EPOXY NANO COMPOSITES: OPTIMIZATION BY TAGUCHI APPROACH"** submitted by me to the Department of Applied Chemistry, Delhi Technological University, Delhi in the partial fulfillment of the requirement for the award of the degree of Master of Technology, is original and not copied from any source without proper citation. This work has not previously formed the basis for the award of any degree, Diploma Associate-ship, Fellowship or other similar title or recognition.

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Date:

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CERTIFICATE

I hereby certify that the project dissertation titled "**STUDIES ON MILLING OPERATIONS ON GRAPHENE OXIDE-EPOXY NANO COMPOSITES: OPTIMIZATION BY TAGUCHI APPROACH"** submitted by Hitesh Goel (Roll No.: 2K15/PTE/12), Department of Applied Chemistry, Delhi Technological University, Delhi in partial fulfilment of the requirement for the award of the degree of Master of Technology, is a record of the project work carried out by him under my supervision. To the best of my knowledge this work has not been submitted in part or full for any Degree or Diploma to this University or elsewhere.

Place: Delhi **Dr. Raminder Kaur**

Date: **SUPERVISOR**

Abstract

This project presents the use of Taguchi optimization methodology in optimizing the cutting parameters of an end-milling process for machining the Graphene Oxide reinforced epoxy hybrid composite material under dry condition. The machining parameters which are been assessed in this analysis are the depth of cut (d), cutting speed (S) and feed rate (f). While the reaction variables to be estimated are the surface roughness of the machined composite surface and the cutting force. A symmetrical exhibit of the Taguchi strategy was set-up and used to dissect the impact of the milling parameters on the surface roughness, material removal rate and cutting force.

The outcome from this examination demonstrates that the use of the Taguchi method can decide the best mix of machining parameters that can give the ideal machining reaction conditions which are the lowest surface roughness and most minimal cutting force value. For the best surface finish, A1-B3-C3 (d = 0.4 mm, S = 1500 rpm, f = 60 mm/min) is observed to be the optimized combination of levels for all the three control factors from the analysis.

In the interim, the streamlined blend of levels for all the three control factors from the analysis which gives the lowest cutting force was observed to be A2-B2-C2 (d = 0.6 mm, S = 1000 rpm, f = 40 mm/min).

Acknowledgement

It gives me incredible delight to express my profound feeling of appreciation and true thanks to the exceptionally regarded Professor Dr. Raminder Kaur for her priceless exhortation, consolation, and help with finishing this work. Supportive recommendations for this work and agreeable conduct are truly perceived.

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Chapter 3: (Materials and Methods) to portray the materials and technique required for the synthesis of the Graphene oxide alongside nano composite of the epoxy-GO, at that point operation done on the milling machine for optimization by Taguchi technique.

Chapter 4: (Results and Discussion) in this Chapter diverse after effects of portrayal and their hypothesis have been investigated.

Chapter 5: (Conclusions) gives fundamental yields of the research work.

Chapter 6: (Future Scope) talks about the various unexplored areas of future applicatins.

Chapter 1

Introduction

Graphene is a single layer of unadulterated carbon; it is firmly pressed layer of carbon atoms which is reinforced together in a hexagonal cross Chapter like a honeycomb. As such it is an allotrope of carbon in the structure of a plane of sp^2 reinforced particles having a bond length of 0.142 nm. Layers of graphene stacked on each other on the best shape graphite. These layers have interplanar separating of 0.335 nm[2].

Low thickness composites and high-quality base are for the most ideal for different applications on the planet. Interesting properties, for the sample, high unbending nature and particular quality, high mechanical quality, high padding, great erosion obstruction and low thermal expansion of the fiber reinforced composite materials have permitted its utilization in the automotive, machine apparatuses, aviation, sports items.

However, the milling of the composites is very difficult to work due to their non-homogenous, anisotropic and reinforced by highly abrasive materials[4]. The composite work piece can cause huge harm and a high wear rate of the cutting instrument. All things considered, the machining of composites relies upon different conditions, for the sample, the properties of the material[5], the relative content of the reinforcement and the matrix material and the reaction to the machining procedure.

Hybrid polymer composites

In polymeric materials, the hybrid is sustained to be a type of reinforced material that is fused into a blend of various grids, or at least two strengthening materials and fillers introduce in a solitary network or the two methodologies. At the point when a material has in excess of two strengthening stages, the fortification of these two stages may prompt positive or negative deviations of the properties anticipated by the blend lead and all the deviations are collectively called as hybrid effects.

In this proposition, the hybrid polymer composite is made of Graphene Oxide[6]. Graphene Oxide has the attributes of good thermal stability, which makes it hard to break development and enhance the quality and strength of the composites of the epoxy matrix. Fusing GO into crossover composite materials should be termed as the new hybrid epoxy composites that have tremendous potential to aid different applicabilities, for the sample, the utilization of quick machining, aviation, defense and automotive. A wide grouping of the composite as matrix and reinforcement[7] is given in Fig. 1.1.

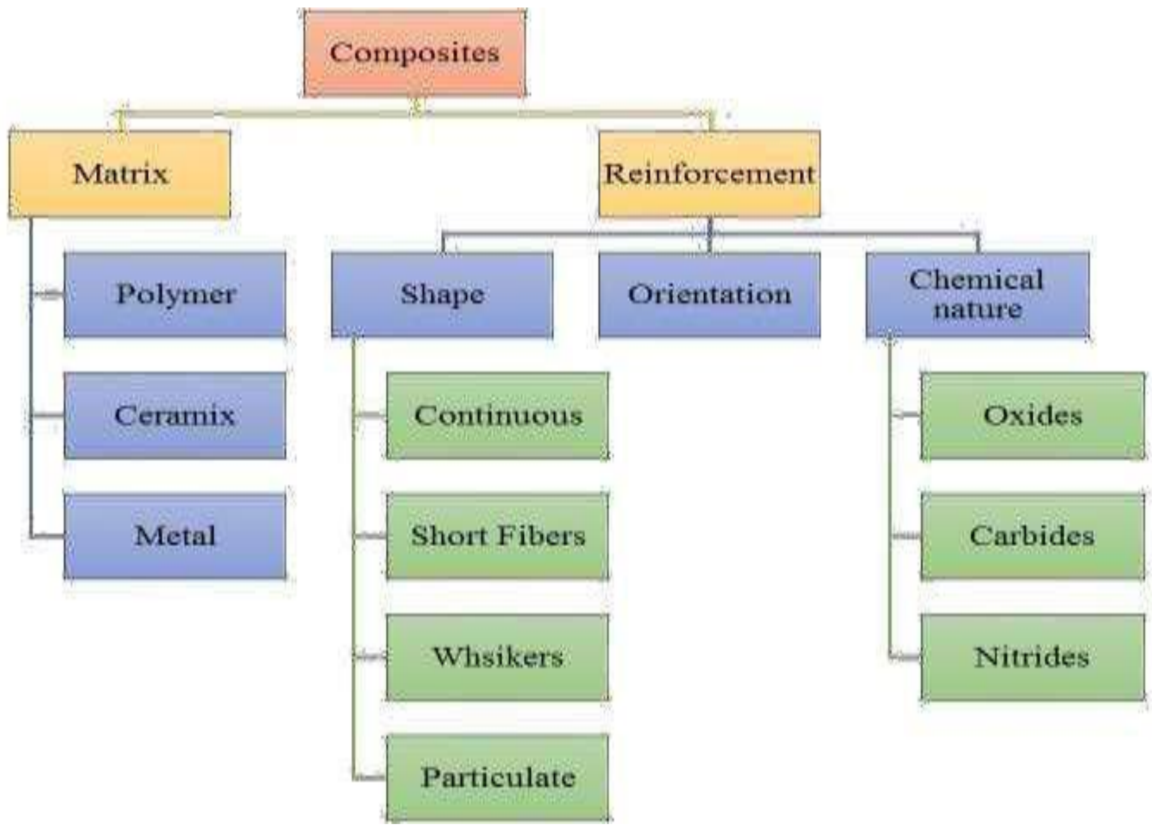


Fig.1.1 Expansive grouping of the composite as matrix and reinforcement

Taguchi method

The test configuration is an intense study device for demonstrating and breaking down the impact of yield execution control factors[8]. The conventional test configuration is hard to utilize, particularly with regards to an extensive number of trials and when the quantity of preparing parameters is expanding. The most vital perspective in the stage plan of the study focuses on the determination of control factors.

Along these lines, the Taguchi method[9], created by Dr. Genichi Taguchi, is presented as an
exploratory procedure that permits to decrease the test number by utilizing orthogonal matrices and limiting the impacts of control parameters. Taguchi is a technique that gives an arrangement of studys to catch information in a controlled way by playing out these tests and information investigates keeping in mind the end goal to pick up data about the conduct of the given process[10]. Further, it is a set of approachs that the natural inconstancy of materials and the generation procedure amid deer outline. It is relatively like the outline of the test (DOE), however, the adjusted test (symmetrical) mix of the Taguchi configuration gives the most proficient partial factorial plan innovation. This procedure has been connected in the production procedures to comprehend the most confounding issues, particularly to watch the level of impact of the control factors.

In Taguchi's definition, the nature of an item is characterized as far as loss of the item provided to the organization from the minute it is sent to the user. Misfortunes because of utilitarian variety are known as the misfortunes because of the deviation of the practical qualities of the item from its wanted target esteem. What's more, clamor factors are the wild factors that reason the utilitarian attributes of an item that does not achieve its particular qualities. The noise factor can be named outer variables (temperature and human mistake), fabricating flaws and disintegration of the item. The primary motivation behind quality designing is to guarantee that the item can be vigorous concerning all conceivable noise factors. Along these lines, the Taguchi strategy could lessen process duration or item encounter, diminish costs by expanding benefits and deciding critical factors in a small span of time, as it can guarantee the nature of the item.

The system of the Taguchi configuration as appeared in Fig. 1.2 can be grouped into three stages in a particular framework configuration, plan parameters and tolerance[11]. The plan parameters considered the most critical stage, can decide the elements that impact the quality attributes in the generation process. The initial phase in the outline of the Taguchi parameters is to choose the proper orthogonal matrix (OA) as indicated by controllable variables. Hence, the analyses were done based on already settled OA and the trial information was broke down to distinguish the ideal condition. Once the ideal conditions distinguished, the affirmation tests are completed with the distinguished ideal levels everything being equal.

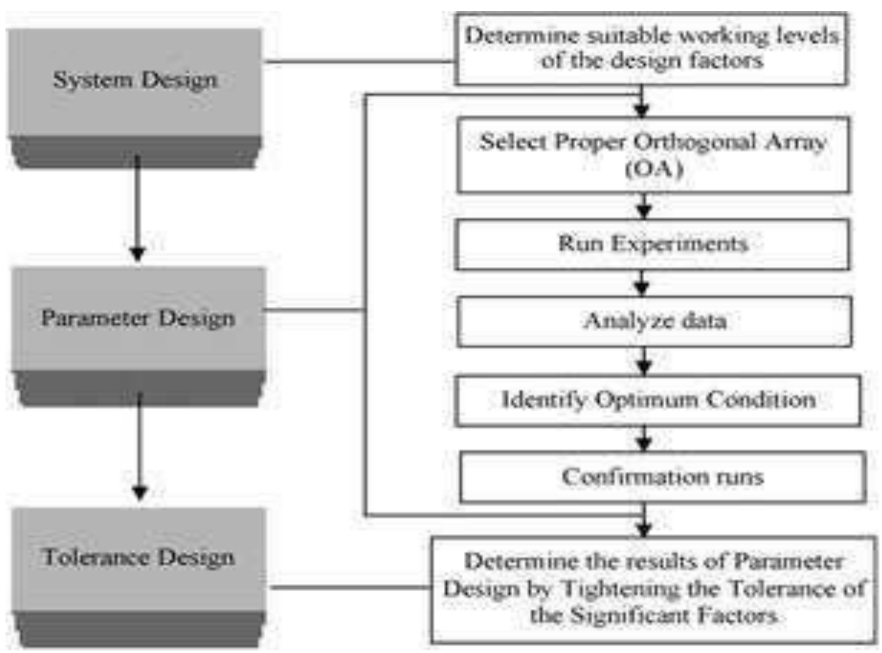


Fig. 1.2 Outline system of Taguchi

The fundamental goal is quality building to deliver strong items for all noise variables. So, Taguchi made a structured orthogonal arrangement to oblige however many variables as could be allowed in the control factor determination procedure to recognize non-critical factors on the main event. Taguchi has utilized the signal-to-noise ratio (S/N) as a quantifiable estimation of the decision of value characteristics[12]. This demonstrates designing frameworks can act in a manner that the elements of creation can be controlled into three classes:

- Control factors, (variables that influence the procedure changeability as estimated by the S/N ratio).
- Signal variables (variables which don't impact the S/N proportion or process mean).
- Factors (variables which don't influence the S/N proportion or process mean).

Objective

In this research, Taguchi outline parameter stage is the biggest plan stage and served to decide the completing fine parameters for acquiring lower surface roughness value and cutting forces value for the GO-Epoxy composite with various milling parametric conditions. The accompanying is the issues contemplated in this experimentation:

- The connection among control factors (cutting profundity, axle speed, and feed rate) what's more, yield reaction factors (surface roughness and shear force).
- Optimum conditions for completing parameters for roughness and cutting strength

Workplan

The making arrangements for the undertaking is done as takes after:

- Synthesis of Graphene oxide
- FTIR and XRD examination for the Graphene oxide
- GO-Epoxy Nanocomposite fabrication
- Milling operations on the sample
- Taguchi analysis

Chapter 2

Literature Review

Until the point when the 1980s, there were just two surely understood individuals from carbon family[13], i.e., graphite and diamond. An awesome unrest has accompanied the revelation of other carbon allotropes, viz., fullerenes, CNTs and as of late graphene. Among all these carbonaceous material, graphene has pulled in colossal research enthusiasm because of its novel, extraordinary properties and in addition basic highlights. Graphene has demonstrated an assortment of captivating properties including unrivaled mechanical properties (inherent quality of ~130 GPa), finish impermeability to any gases, capacity to maintain to a great degree high densities of electric current (a million times higher than copper), high electron portability at room temperature (250,000 cm^2/Vs), uncommon warm conductivity

(5000 $Wm^{-1} K^{-1}$) and simple chemical functionalization comparative to different materials (Table-1[14]. Additionally, the cost of creation of graphene is low when contrasted with other carbon-based nanomaterials.

Table 2.1: Graphene, CNT, nano-sized steel and other polymers various properties

Materials	Tensile strength (GPa)	Thermalconductivity (W/mK)	Electrical conductivity (S/m)
Graphene	160±10	$(4.84\pm0.44)\times10^3$ to $(5.30\pm0.48)\times10^3$	7200
CNT	60-150	3500	3000-4000
Nano sized steel	1769	5-6	1.35×10^6
Plastic (HDPE)	0.018-0.020	0.46-0.52	Insulator
Rubber (Natural)	0.02-0.03	0.13-0.142	Insulator
Fiber (Natural)	3.62	0.04	Insulator

2.1 Foundation of Carbon Materials

At first, carbon was found by A.L. Lavoisier as charcoal[15] in 1789. It is broadly dispersed in nature with coal as its primary source. Carbon has 4 electrons in four hybridized holding orbital ($2s^1 2px^1 2py^1 2pz^1$) that are accessible to shape covalent bonds. It is being produced into various allotropes, viz., diamond, graphite, fullerenes, carbon nanotubes and all the more of late graphene. These allotropes demonstrate an assortment of fascinating electronic and auxiliary properties because of the conjunction of sp^2 furthermore, sp^3 hybridized carbon atoms in various extents.

2.2 Graphite

Graphite is a delicate material in which carbon particles are reinforced trigonally with three carbon atoms in the plane as appeared in fig 3. Because of such sort of bonding, we expect a two-dimensional layer compose engineering has sp^2 hybridization. Because of intermolecular communications, these layers stack over each other to frame 3D graphitic structure. These layers are reinforced by frail Vander Walls forces in charge of the delicate quality of graphite and in this manner indicate greasing up properties. The **delocalization of one of the alternate electrons of every atom shapes a π**-cloud because of which graphite is an electrical conductor[16]. The interlayer distance between layers is observed to be 3.34Å, with **ABAB…. stacking sequence.**

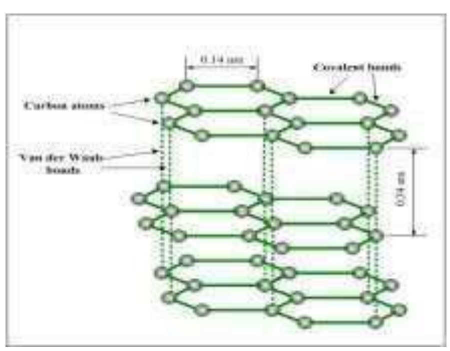


Figure 2.1: General structure of graphite

2.3 Diamond

Diamond is an allotrope of carbon with sp^3 hybridized orbitals which are organized in 3D space to frame tetrahedral structure[17] as appeared in fig 2.2. Diamond is the hardest known material due to solid covalent holding. All the orbitals in diamond are loaded with no free electron in valence shell because of which diamond is a protecting material with wide band hole. The majority of the utilizations of diamond are identified with a modern reason like granulating, boring, cleaning and slicing because of its unparallel hardness. Other than its appropriate refractive record, high optical scattering and capacity to cut along different diamond planes give the jewel its trademark brilliance which makes it valuable gemstone for adornments and trimmings.

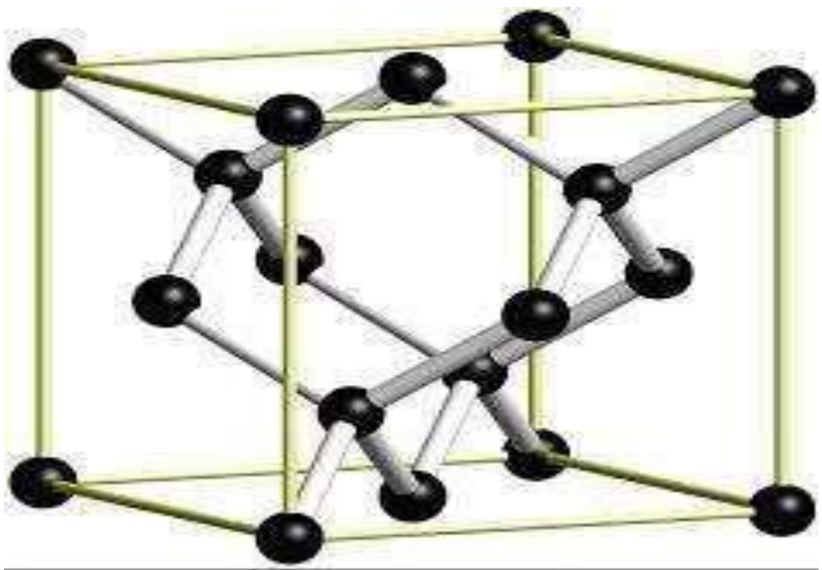


Figure 2.2: General structure of diamond

2.4 Fullerene

A fullerene is an allotrope of carbon which has numerous structures, for the sample, empty circle, tube and numerous different shapes. Round fullerenes, likewise alluded to as Buckminster fullerenes or buckyballs. Fullerenes are like graphite is made of stacked graphene sheets of the connected hexagonal ring, yet they may likewise have either pentagonal or heptagonal rings[18] as appeared in fig 2.3. Buckyballs are liable to serious research, both for their special science and for their innovative applications, particularly in nanotechnology, hardware, and material science, e.g., as electron acceptor on photovoltaic, for making incorporation mixes, quantum mechanics considers, super conductivity.

Figure 2.3: General structure of fullerene

2.5 Carbon Nanotubes

Carbon nanotubes[19] (CNTs) are allotropes of carbon which have beamrel shaped nanostructure as appeared in Fig 2.4. CNTs are developed with the length-to-measurement proportion of up to 135,000,000:1, impressively bigger when contrasted with some other material. Due to their, astonishing mechanical, electrical and thermal properties, carbon nanotubes are utilized as added substances to different basic assets. For instance, CNTs are utilized to fabricate petite bits of the material(s) in a few (basically carbon fiber) golf clubs, bats or vehicle parts. The graphene sheets are moved at particular and discrete edges, and the blend of the moving edge and sweep chooses the nanotube properties; for instance, regardless of whether the individual nanotube shell is a metal or semiconductor. CNTs are separated as single-walled nanotubes (SWNTs) and multi-walled nanotubes (MWNTs). Singular nanotubes normally adjust themselves into "ropes" which are held together by **Vander Waals forces, all the more particularly, π**-stacking.

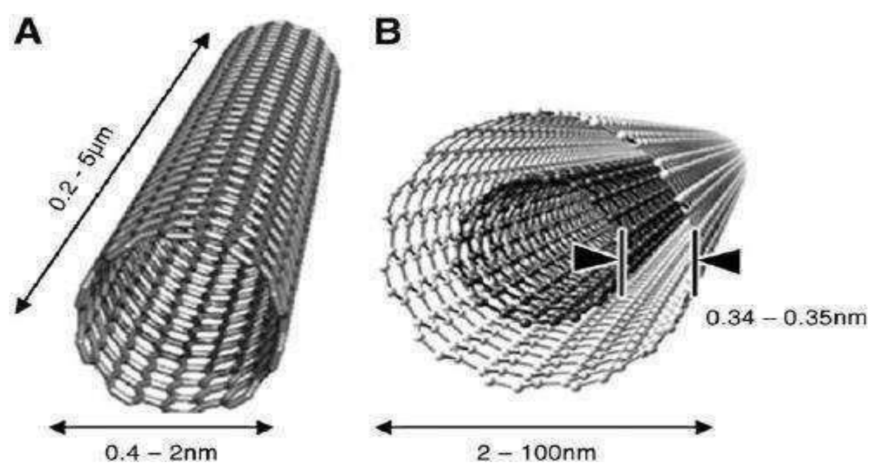


Figure 2.4: Structure of (a) single wall and (b) multi-walled CNT

2.6 Graphene

The term graphene first showed up in 1987 to portray single sheets of graphite as one of the constituents of graphite intercalation compounds[19] (GIC). Thoughtfully, a GIC is a crystalline salt of the intercalant and graphene. The term was additionally utilized as a part of early portrayals of carbon nanotubes and in addition for epitaxial graphene. Graphene is a one-particle thick planar sheet of sp^2 reinforced carbon particles which are thickly stuffed in a honeycomb molded cross Chapter. In a 2-D carbon framework for graphene, three carbon electrons from four hybridized holding electrons shape solid in-plane sp^2 bonds comprising of the honeycomb structure, furthermore, a fourth electron spreads out finished the best or base of the layer as a π electron[20].

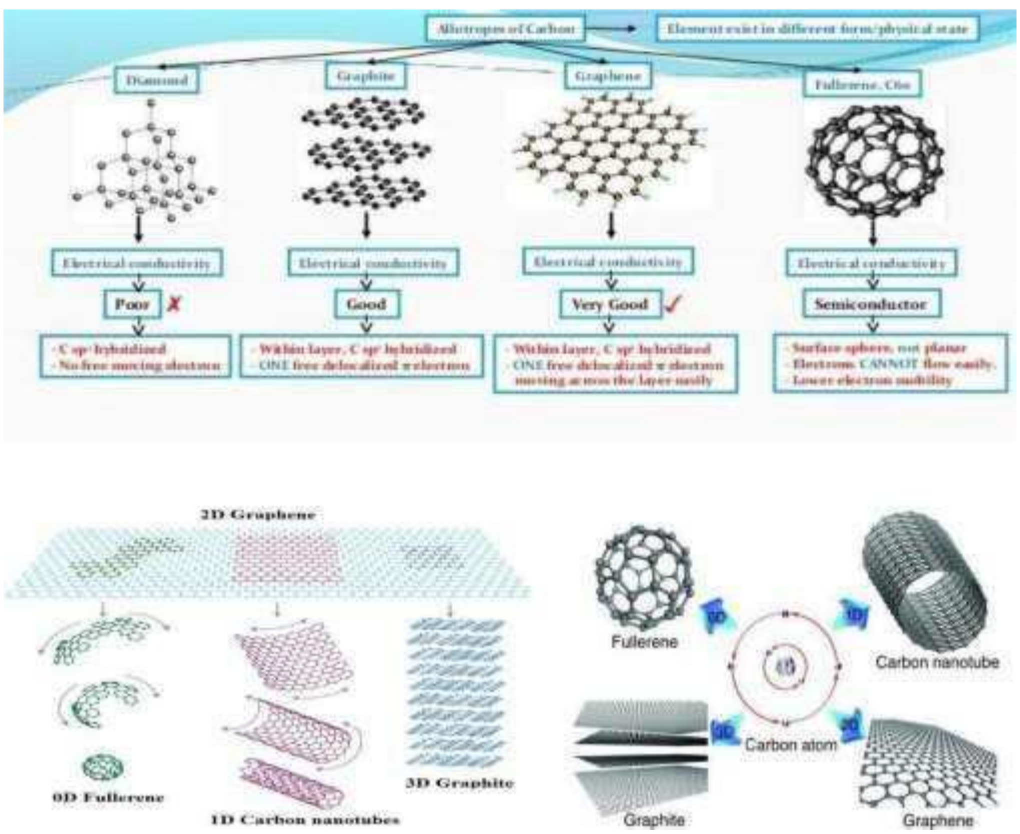


Figure 2.5: Graphene and its connection to fullerene, CNT, and graphite

2.7 Fourier Transform infrared spectroscopy (FTIR)

FT-IR remains for Fourier Transform InfraRed, the favored technique for infrared spectroscopy. In infrared spectroscopy, IR radiation is gone through a sample[21]. A portion of the infrared radiation is consumed by the sample and some of it is gone through (transmitted). The subsequent range speaks to the atomic ingestion and transmission, making a sub-atomic unique finger impression of the test. Like a finger impression, no two one of a kind atomic structures creates a similar infrared spectrum[22]. This makes infrared spectroscopy helpful for a few kinds of examination. In this way, what data can FT-IR give?

- It can recognize obscure materials
- It can decide the quality or consistency of a sample
- It can decide the measure of segments in a blend

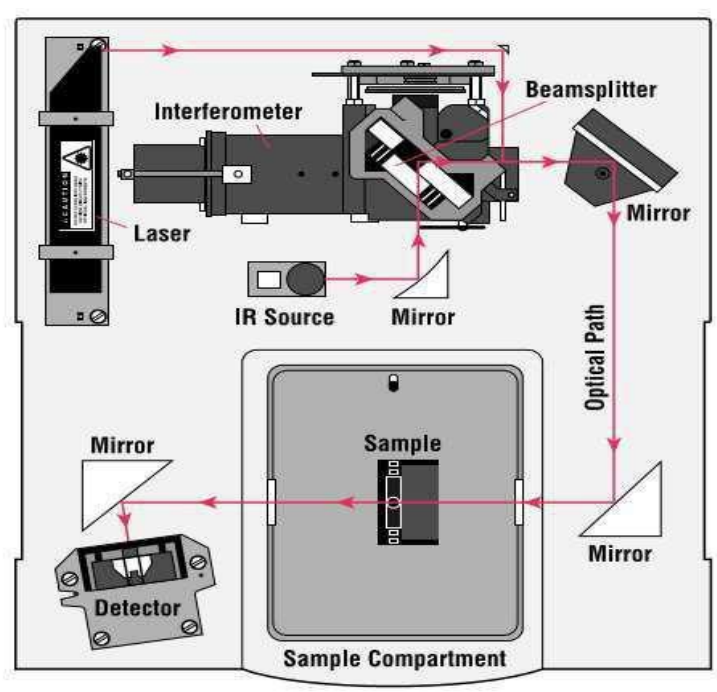


Fig. 2.6 Provision of a spectrophotometer

Analysis process of FTIR

The ordinary instrumental process is as per the following:

1. The Source: Infrared energy is discharged from a shining dark body source. This beam passes through an opening which controls the measure of energy introduced to the sample (and, at last, to the identifier).

2. The Interferometer: The beam enters the interferometer where the spectral encoding takes place. The subsequent interferogram flag at that point leaves the interferometer.

3. The Sample: The beam enters the sample compartment where it is transmitted through or reflected off the surface of the sample, contingent upon the sort of study being refined. This is the place of particular frequencies of energy, which are interestingly normal for the sample, are consumed.

4. The Detector: The beam at long last goes to the identifier for definite estimation. The identifiers utilized are extraordinarily intended to gauge the exceptional interferogram flag.

5. The Computer: The deliberate flag is digitized and sent to the PC where the Fourier change happens. The last infrared range is then displayed to the user for translation and any further control and can be found in the Fig.2.7.

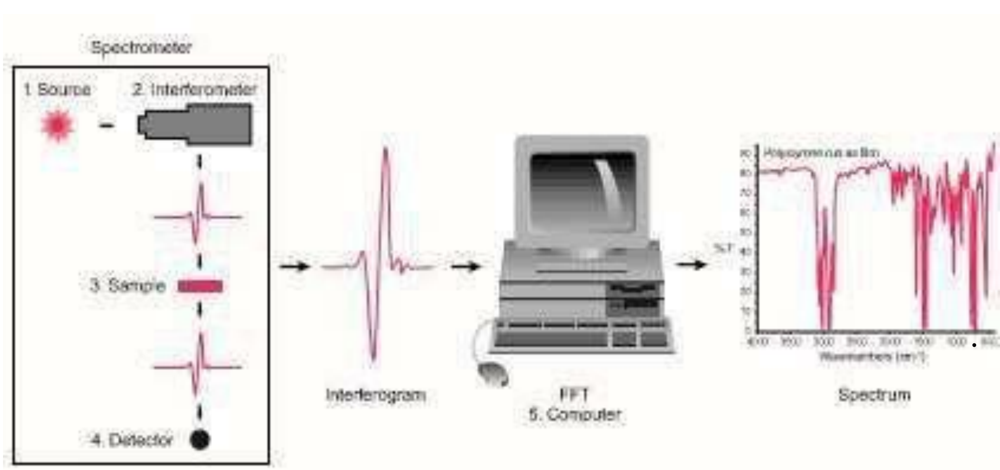


Fig.2.7: The sampling method for FTIR

2.8 Scanning Electron Microscopy (SEM)

A scanning electron microscope[2] (SEM) is a kind of electron magnifying instrument that produces pictures of a sample by examining the surface with an engaged light emission. The electrons communicate with particles in the sample, delivering different signs that contain data about the sample's surface geography and piece. The electron pillar is checked in a raster filter design, and the beam's position is joined with the identified flag to deliver a picture. SEM can accomplish determination superior to 1 nanometer. Samples can be seen in high vacuum in traditional SEM, or in low vacuum or wet conditions in factor weight or ecological SEM, and at an extensive variety of cryogenic or raised temperatures with specific instruments. The most widely recognized SEM mode is the discovery of optional electrons discharged by atoms energized by the electron pillar. The quantity of auxiliary electrons that can be recognized depends, among other things, on sample geography. By examining the sample and gathering the optional electrons that are produced utilizing an extraordinary indicator, a picture showing the geology of the surface is made.

Scanning process and image formation

In a run of the mill SEM, an electron beam is thermionically transmitted from an electron weapon fitted with a tungsten fiber cathode[24]. Tungsten is typically utilized as a part of thermionic electron guns since it has the most elevated liquefying point and least vapor weight everything being equal, consequently enabling it to be electrically warmed for electron outflow, and in the light of its ease. Different kinds of electron producers incorporate lanthanum hexaboride (LaB_6) cathodes, which can be utilized as a part of a standard tungsten fiber SEM if the vacuum framework is updated or field emission guns (FEG), which might be of the chilly cathode compose utilizing tungsten single crystal producers or the thermally helped Schottky compose, that utilization producers of zirconium oxide[25].

The electron beam, which regularly has an energy extending from 0.2 keV to 40 keV, is engaged by maybe a couple condenser focal points to a spot around 0.4 nm to 5 nm in width. The beam passes through sets of checking loops or combines of diverter plates in the electron segment, ordinarily in the last focal point, which avoid the beam in the x and y tomahawks. The SEM that is utilized as a part of general research facilities can be found in Fig 2.8.

Fig.2.8: The pictorial perspective of SEM machine

2.9 X-ray diffraction (XRD)

X-ray Diffraction is a method utilized for deciding the nuclear and sub-atomic structure of a crystal, in which the crystalline particles cause a light emission X-rays to diffract into numerous particular headings. By estimating the points and forces of these diffracted beams, a crystallographer can deliver a three-dimensional photo of the thickness of electrons inside the crystal. From this electron thickness, the mean places of the molecules in the crystal can be decided, and in addition to their substance bonds, their turmoil, and different other data. Since numerous materials can frame crystals, for example, salts, metals, minerals, semiconductors, too as different inorganic, natural, and organic atoms—X-ray crystallography has been essential in the advancement of numerous logical fields. In its first many years of utilization, this technique decided the extent of molecules, the lengths, and sorts of substance bonds, and the nuclear scale contrasts among different materials, particularly minerals and compounds.

The technique likewise uncovered the structure and capacity of numerous organic particles, including vitamins, medications, proteins and nucleic acids, for example, DNA. X-ray crystallography is as yet the main strategy for describing the nuclear structure of new materials and in observing materials that seem comparative by other tests. X-ray crystal structures can likewise represent surprising electronic or flexible properties of a material, shed light on synthetic communications and procedures, or fill in as the reason for planning pharmaceuticals against ailments.

X-ray crystallography demonstrates the course of action of water particles in ice, uncovering the hydrogen bonds in Fig. 2.9 that hold the strong together[26]. Barely any different techniques can decide the structure of matter with such accuracy.

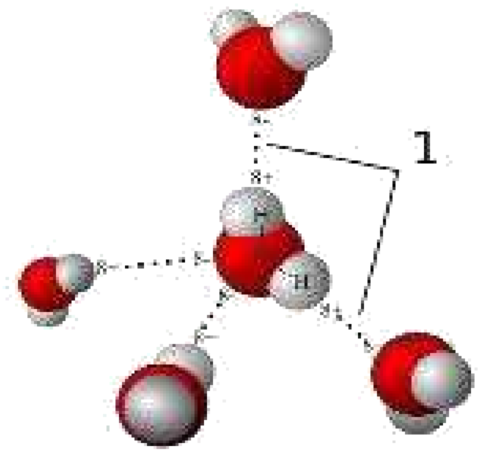


Fig. 2.9: The course of action of water molecules in ice

X-beam crystallography is identified with a few different strategies for deciding nuclear structures. Comparable diffraction samples can be created by dissipating electrons or neutrons, which are in like manner translated by Fourier change. In the event that solitary crystals of adequate size can't be acquired, different other X-beam strategies can be connected to get less nitty gritty data; such techniques incorporate fiber diffraction, powder diffraction and (if the sample isn't solidified) small angle X-beam disseminating (SAXS). In the event that the material under scrutiny is just accessible in the frame of nanocrystalline powders or experiences poor crystallinity, the techniques for electron crystallography can be connected for deciding the nuclear structure.

2.10 Milling Machine

Figure 2.10: The pictorial perspective of the milling machine

Milling is the machining procedure of utilizing rotating cutters to expel material from a workpiece by progressing (or bolstering) toward a path at a point with the pivot of the instrument. It covers a wide assortment of various activities and machines, on scales from little individual parts to expansive, substantial posse milling tasks. It is a standout amongst the most normally utilized procedures in industry and machine shops today to machine parts to exact sizes and shapes. Milling should be possible with an extensive variety of machine tools[27].

The first class of machine apparatuses for milling was the milling machine (regularly called a factory). After the approach of PC numerical control (CNC), milling machines developed into machining focuses (milling machines with programmed device changers, device magazines or merry go rounds, CNC control, coolant frameworks, and fenced in areas), by and large, delegated vertical machining focuses (VMCs) and flat machining focuses (HMCs). The reconciliation of milling into transforming conditions and of transforming into milling conditions started with live tooling for machines and the infrequent utilization of plants for turning operations[28], prompted another class of machine instrument, multitasking machines (MTMs), which are reason worked to accommodate a default machining system of utilizing any blend of milling.

2.11 Taguchi Analysis

Distinctive machining forms were optimized by the specialists to enhance the nature of the instrument G.Akhyar et al[29]. connected Taguchi enhancement strategy for the improvement of cutting parameters in turning Ti-6%, Al-4% with covered and uncoated established carbide devices under dry conditions and high cutting rates for better surface unpleasantness. An L27 symmetrical cluster including four factors, for example, Cutting rate, feed rate, depth of cut and apparatus grades with three levels for each factor was utilized to distinguish the ideal blend. They utilized ANOVA to decide the slicing pace and device review to be a huge factor that influenced the surface complete M.Y. Noordin et al. utilized RSM to consider the execution of a multilayer tungsten carbide instruments while turning AISI 1045 steel with the steady profanity of cut and feed rate.

To think about the impacts of three factors, for example, cutting velocity, feed and side bleeding edge (SCEA) on surface roughness and the distracting force utilizing face focused CCD. There are different variables that give auxiliary commitments to the execution markers. On account of surface unpleasantness, the SCEA2 and the collaboration of feed and SCEA gives these commitments while to extraneous force, the SCEA2, the communication of feed and SCEA; and the cutting pace gives them. Hari Vasudevan et al. utilized Taguchi strategy to streamline the milling parameters on Glass Fiber Reinforced Plastic NEMA 11[30]. In this study, an endeavor has been made to streamline milling parameters with various execution attributes, in view of the Gray Relational Analysis combined with Taguchi strategy. The milling tests were completed on a vertical Milling machine.

The tests were led by L18 (OA). The four cutting parameters chose for this examination are milling methodology, axle speed, feed rate and depth of cut. Mohammed T. Hayajneh et al. assemble a different relapse display for surface roughness to ponder the impacts of 30 shaft speed, cutting feed rate, depth of cut and their two way connections.

The cutting parameters were chosen as four levels of cutting velocity, seven levels of feed rate and three levels of depth of cut. The outcomes demonstrated the cutting feed as the most overwhelming component and communications cutting feed-depth of cut and cutting feed-axle speed the most critical. Ghhani J. A. et al. optimized cutting parameters in end milling process while machining solidified steel AISI H13 with TiN covered P10 carbide embed apparatus under semi-completing and completing states of high speed cutting[31].

The impact of milling parameters, for example, cutting rate, feed rate and depth of cut alongside their connections on the procedure are examined utilizing the Taguchi technique for the exploratory plan (DOE). The consider showed the reasonableness of Taguchi strategy to tackle the expressed issue with least number of preliminaries as contrasted and a full factorial plan. Shreemoy Kumarnayak et al.

The Study has completed the impact of machining parameters amid dry turning of AISI 304 austenitic spotless steel[32]. For this study HMT, hard core machine was utilized. They have embraced L27 symmetrical cluster with three machining parameters like cutting velocity, feed rate and depth of cut and three significance attributes of machinability, for example, material expulsion rate (MRR), cutting force and surface roughness (R_a) were estimated.

Chapter 3

Materials and Methods

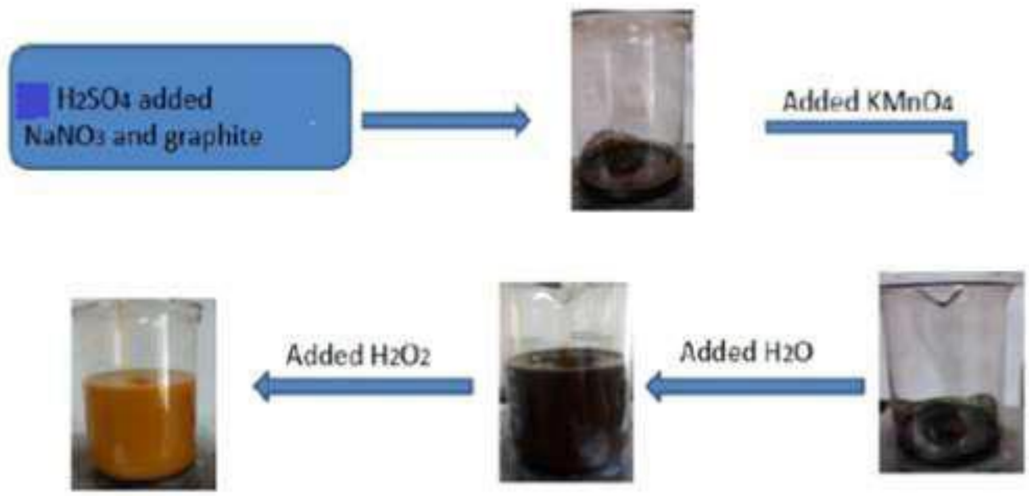
3.1 Raw Materials

- Graphite powder (CHD, India)
- Sodium nitrate $NaNO_3$ (Fischer scientific)
- Potassium permanganate $KMnO_4$ (Fischer scientific)
- Sulphuric acid H_2SO_4, 98% (Merck chemicals)
- Hydrogen peroxide H_2O_2, 30% (Speck pure chemicals)
- Epoxy
- TETA (as a hardener)

3.2 Synthesis of GO

- To prepare GO utilizing Hummer's Method[33], we took a spotless dry measuring glass and flushed it using concentrated sulphuric acid.
- Took 75ml of H_2SO_4 and after that added 3gm of $NaNO_3$. Stirred this solution for 1 h, at that point added 3gm of graphite and stirred for 3 h.
- We added 9gm of $KMnO_4$ gradually while keeping it on an ice shower so that the temperature of the solution does not transcend 20 C.
- Stirred this solution on a magnetic stirrer for 24 h.

- Then added 150 ml of water to this solution and stirred for 20 min and after that added 420ml of water and stirred for 10 min.
- Then we added H_2O_2 to the solution till a splendid yellow shading is gotten.
- Then filtered the solution and washed the precipitate with water 2-3 times or till all the corrosive is evacuated. Centrifuged the solution and the GO pulp is expelled.
- We put the GO pulp in the broiler for 5-6 h and gather the powder in eppendorf.



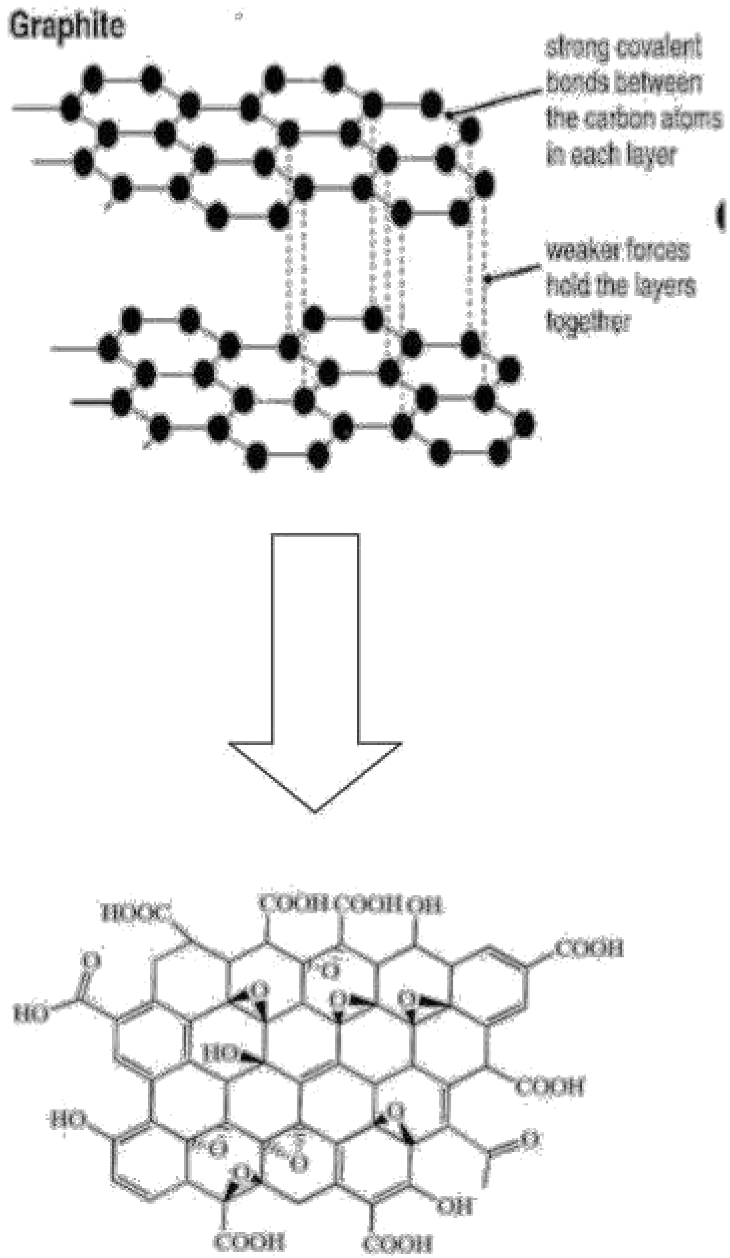


Figure 3.1: Oxidized type of Graphite

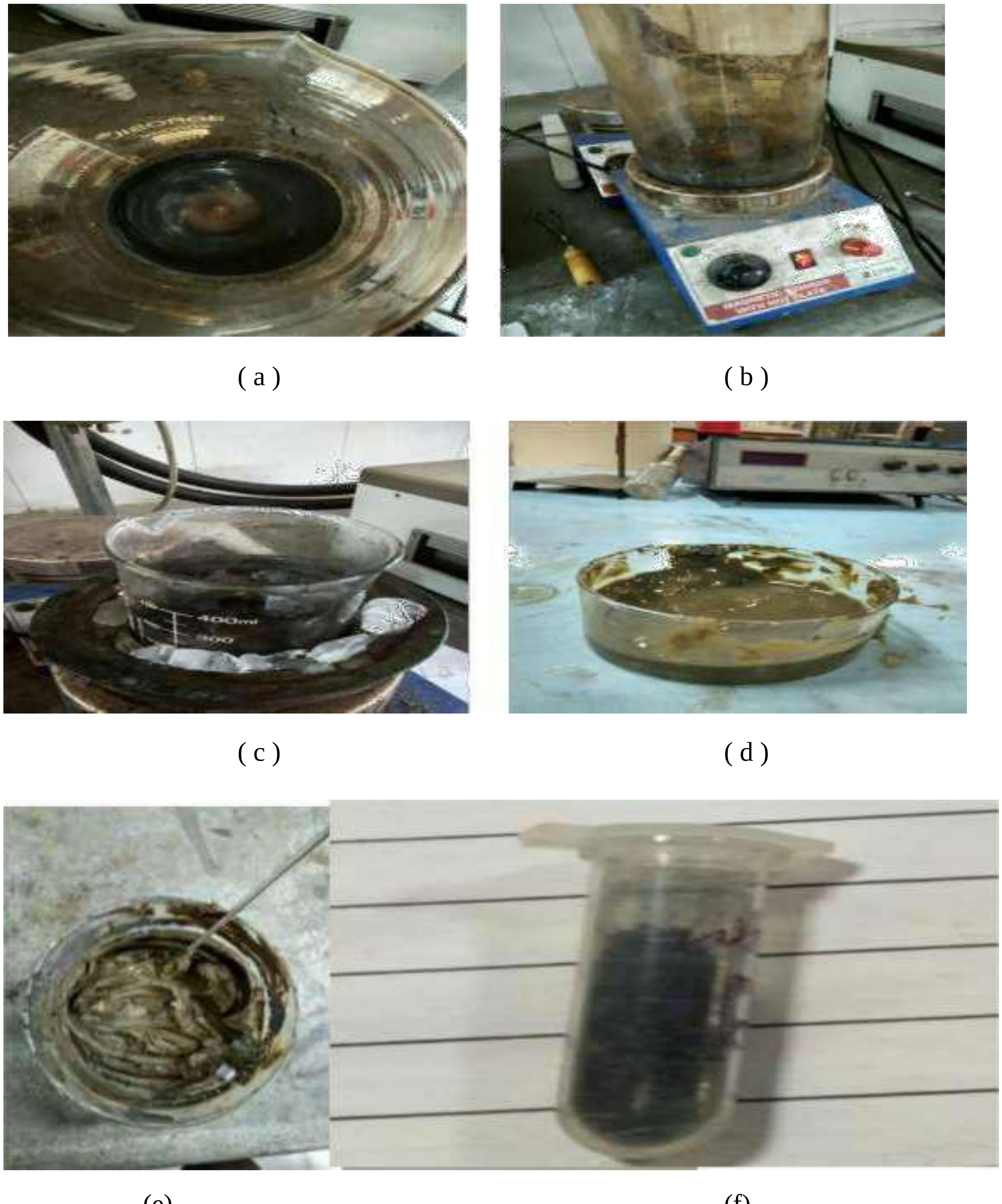


Figure 3.2: The different stages of the synthesis GO (a) and (b) solution on the magnetic stirrer after addition of graphite (c) solution on the ice bath (d) the pulp after centrifugation (e) the pulp after nanocomposite 5 h (f) GO in eppendrof in powder form.

3.3 Sample Preparation

• Clean the mold and dry it totally.

• Weigh 100 g of epoxy resin and pour it in a measuring glass.

• Weigh 1 g of hardner and pour it in an alternate measuring glass.

• Weigh graphene oxide as indicated by wanted fixations (1% of 110 g)

• Mix graphene oxide with epoxy resin took after by hardener.

• Finally, with the assistance of brush pour the blend of resin, hardner and GO over the shape.

• Make beyond any doubt that shape surface is covered with discharge operators like Vaseline for simple evacuation of the test.

• Keep the sample for 24 h for curing.

• After curing is done, securely take the sample out.

• Sample measurement: 114x88x8 mm^3

(a) (b)

(c)

(d)

Fig 3.3. Diverse phases of sample preparation (a, b, c, d)

3.4 Milling Operation

- The sample was machined in the project shop of the mechanical engineering department.
- The sample was first made prepared to clasp on the milling machine.
- File and clasp were utilized to make the sample appropriate for the milling as appeared in Fig. 16.
- The rectangular sample made up of the GO-Epoxy composite was fitted to a milling machine.
- Slots of width 5 mm were accessible for the working

Fig 3.4: Preparing a sample for the milling machine

- For Taguchi methodology, we require three distinct components for the optimization.
- In this exploration, we are taking feed rate (f), shaft speed (s), and depth of cut (d) as three factors on which improvement should be done.
- Feed rates were taken as 20, 40,60 (mm/min)
- The depth of cut was taken as 0.4, 0.6, 0.8 (mm)
- Spindle speed was 500, 1000, 1500 (rpm)
- 27 spaces were made on the sample of length 20 mm

Figure 3.5: Machining on the neat sample

Figure 3.6: Machining on the GO-epoxy sample

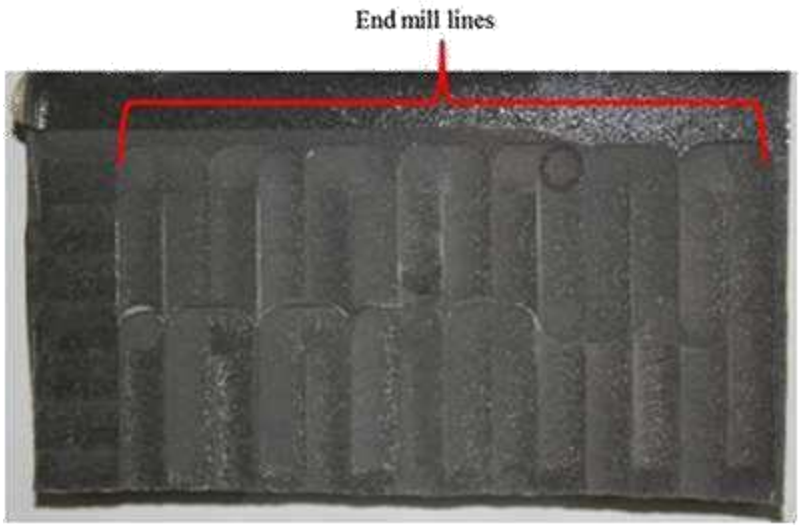


Figure 3.7: Slots cutting on the sample

In this test, twenty seven distinct mixes of processed lines with 6 mm width were made by playing out an end-milling task (dry condition) on the crossover composite example as appeared in fig 3.7. The milling activity forms were performed in milling focus. In the interim, the cutting instrument utilized is the NKO end-milling apparatus with four woodwinds (dia-6 mm). The surface harshness estimation was finished by utilizing a compact surface roughness analyzer TR200 (measures R_a in l m; stylus travel 1.5 mm cut-off).

Other than that, the cutting powers were likewise estimated online amid end-milling activity with a touchy three-part Kistler 5070A compose piezoelectric dynamometer with a charge intensifier. The information procurement was made through the charge intensifier and a PC utilizing the proper software (Dynoware). The microsurface morphology thinks about was done utilizing the Scanning Electron Microscope Hitachi S-3400N.

Chapter 4

Results and Discussion

4.1 Fourier transform infrared spectroscopy

The FTIR spectra have been utilized to gauge, how well the sample retains the light at which
wavelength, by utilizing this we can discover, which functional group is available in the material.

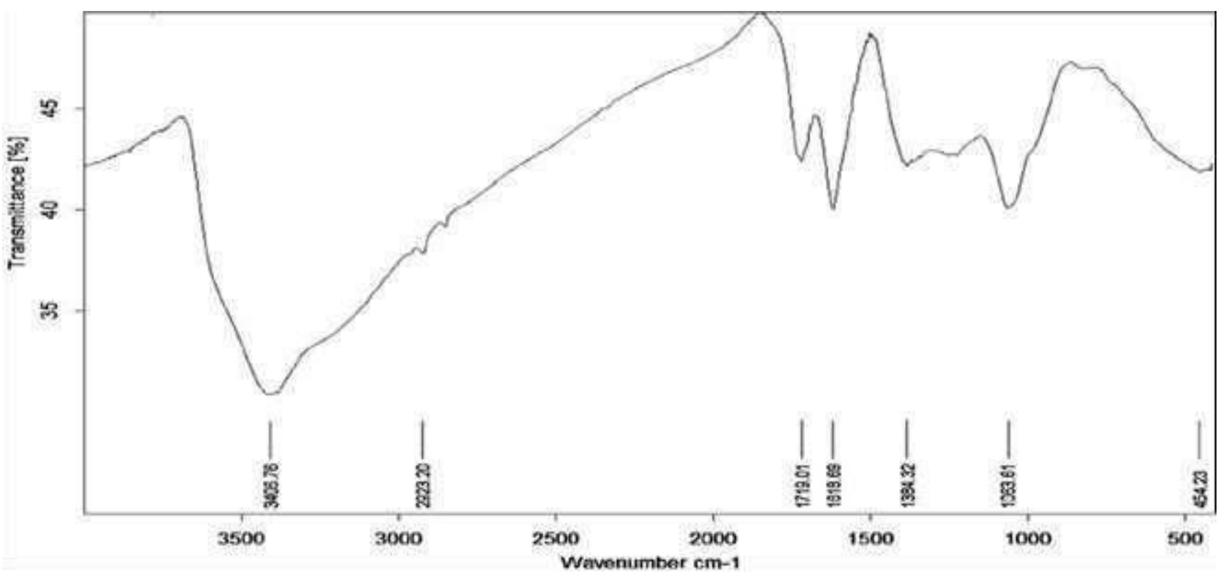


Figure 4.1: FTIR Spectra of Graphite Oxide

GO nanocomposite. A noticeable pinnacle adsorption showed up at 3407 cm^{-1} uncovers the vibration of the hydroxyl group (O-H) exhibit in GO. This OH-bond might be because of the presence of alcohol, phenol, carboxylic acids et cetera.

Because of the nearness of water at this vibration recurrence likewise affirms the arranged hydrophilic properties of GO. Moreover, the upper parts (1696, 1233 and 1064) cm^{-1} of the carbonyl group (C = O), the Epoxy and alkoxy groups. The C = C is available in the 1.627 cm^{-1} band that gives the mark of the carbon groups doesn't oxidize.

4.2 Powder X-Ray Diffraction Analysis

X-beam diffraction is a logical apparatus used to recognize the nuclear and atomic structure of the crystals in which the incident beam is diffused in numerous ways. Taking a gander at the forces and edges of the diffracted shaft can give a 3-D picture of the electronic thickness in the crystal and accordingly utilize this data, we can decide the different substance bonds, a crystal disorder, and considerably more data about the crystal plane. In our present study, we played out the incorporated GO and analyzed the untreated graphite. The XRD spectra are estimated in the scope of 2Θ i.e. to 5, demonstrating a peak of graphite diffraction at a 26 angle is the distance between the layers is then figured utilizing the Brags equation, that is, Sin2d = Nλ.

That's relatively equivalent to 3.34 for graphite. While the oxygen functionality group presented in graphite, there is a development of the graphite layers and the partition between graphite builds, so the diffraction angle must be lower. It is plainly seen in the XRD spectra of GO that the angle of diffraction of GO is at a 12° edge and, along these lines, the partition between the crests is relatively equivalent to 7.97.

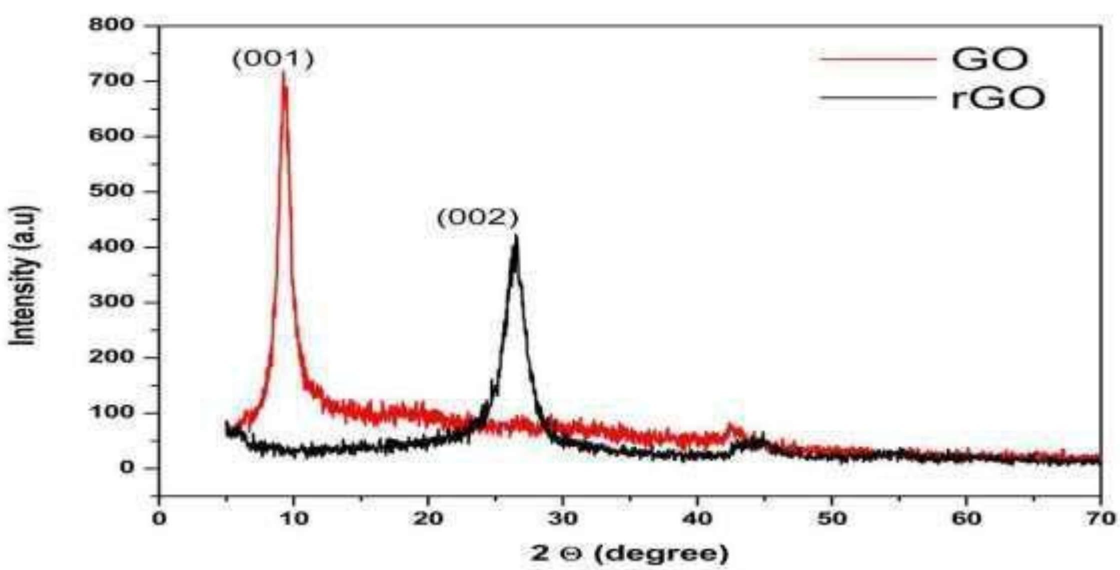


Figure 4.2: Diffraction Pattern of the X-Ray Graphine Oxide (GO) & the reduced Graphene Oxide (rGO)

4.3 TAGUCHI ANALYSIS: Orthogonal Array and Test Variables

In the outline period of the Taguchi technique parameters in figure 2, the initial step is to arrange and select a suitable orthogonal array[34].

Table 4.1 The basic Taguchi $L_{27}(3^3)$ Orthogonal Array.

Run	Control factors and levels		
	A	B	C
1	1	1	1
2	1	1	2
3	1	1	3
4	1	2	1
5	1	2	2
6	1	2	3
7	1	3	1
8	1	3	2
9	1	3	3
10	2	1	1
11	2	1	2
12	2	1	3
13	2	2	1
14	2	2	2
15	2	2	3
16	2	3	1
17	2	3	2
18	2	3	3
19	3	1	1
20	3	1	2
21	3	1	3
22	3	2	1
23	3	2	2
24	3	2	3
25	3	3	1
26	3	3	2
27	3	3	3

Table 4.2 Parameters, codes, and level values utilized for orthogonal array

Parameter	Code	Level 1	Level 2	Level 3
The control factors				
Cutting depth, d (mm)	A	0.4	0.6	0.8
Spindle speed S (rpm)	B	500	1000	1500
Advance, f (mm / h)	C	20	40	60
Response variables				
The surface roughness, Ra (lm)	-	-	-	-
Cutting force, Fr (N)	-	-	-	-

To oblige three control variables into the testing process, an institutionalized Taguchi-based experimental design, L27 was been utilized as a part of this analysis and is appeared in Table 4.1. This fundamental configuration utilizes three control factors with optimum three sub levels each and the plan has the capacity to keep a check on the cooperation between the components. According to the standard plan (Table 4.1), there is 27 trial runs that should be directed with the blend of levels for each control factor (A– C). As the fuse of the noise variables into the OA is discretionary, the clamor factor is discarded from this test think about.

The chose variables are shown in Table 4.1 along with their naming convention and qualities for the applicability in Taguchi parameter design analysis. In this examination, the control variables (Depth of cut, Spindle speed, and Feed rate) are the kind of variables which are independent while the reaction factors (Surface roughness & Cutting forces) are the dependendable variables. In Table 4.3, an altered OA has been made by utilizing fundamental Taguchi OA (Table 4.2) also, the selected parameters from Table 4.1. In this changed OA, the fundamental varieties of control factors are joined with the varieties of reaction factors alongside the S/N proportion (g) qualities and it conveys to the aggregate number of 27 exploratory runs.

Table 4.3. Orthogonal Array

Run	internal control factor matrix A	B	C	R_a	g_{Ra}	F_r	g_{Fr}
1	1	1	1	1.15	1.21	94.31	39.49
2	1	1	2	1.94	5.74	41.54	32.37
3	1	1	3	1.18	1.47	38.87	31.79
4	1	2	1	0.96	0.34	16.44	24.32
5	1	2	2	0.62	4.15	5.45	14.73
6	1	2	3	0.77	2.25	35.85	31.09
7	1	3	1	1.06	0.48	43.78	32.83
8	1	3	2	0.36	8.95	35.46	30.99
9	1	3	3	0.29	10.84	83,02	38.38
ten	2	1	1	1.43	3.12	60.28	35.60
11	2	1	2	0.89	1.01	1.63	4.23
12	2	1	3	1.28	2.14	45.85	-33.23
13	2	2	1	1.10	0.82	12.76	22.12
14	2	2	2	0.86	1.36	7.57	17.58
15	2	2	3	0.77	2.23	23,60	27.46
16	2	3	1	0.37	8.71	9.46	19.52
17	2	3	2	0.63	3.97	4.81	13.64
18	2	3	3	0.84	1.55	47.63	33.56
19	3	1	1	1.14	1.12	3.13	9.92
20	3	1	2	1.80	5.08	62.40	35,90
21	3	1	3	1.81	5.16	93.45	39.41
22	3	2	1	1.15	1.22	41.94	32.45
23	3	2	2	1.43	3.08	2.27	7.11
24	3	2	3	2.11	6.47	42.31	32.53
25	3	3	1	0.95	0.47	8.41	18.50
26	3	3	2	1.25	1.94	23.51	27.42
27	3	3	3	0.47	6.54	14.23	23.06
	A	B	C				
R_a Effects							
Level 1	0.93	1.40	2.92				
Level 2	0.91	1.08	1.08				
level 3	1.34	0.69	1.06				
Ra Effects							
Level 1	1.96	2.67	0.17				
Level 2	1.42	0.14	0.40				
level 3	1.89	4.29	0.91				
F_r Effects							
Level 1	43.86	49.05	32.28				
Level 2	23.73	20.91	20.52				
level 3	32.41	30,03	47,20				
Fr Effects							
Level 1	30.67	29.10	-26.08				
Level 2	22.99	23.27	20.44				
level 3	25.14	26.43	32.28				

Information breaking down on the ideal levels for all control factors is the initial step subsequent to finishing the trial arrange. The fundamental go for this analysis is to improve the milling parameters to acquire bring down surface roughness and ideal resultant cutting force esteems so the littler the better trademark was picked in this examination. The outcomes from the exploratory stages are the surface roughness, R_a and cutting force, Fr esteems are recorded in Table 4. Reasonable S/N approach is prescribed by Taguchi for the methods and S/N proportion analysis which includes charting the impacts and outwardly distinguishing the elements which seem, by all accounts, to be critical without utilizing ANOVA examination. Therefore, these made the way to deal with turn out to be more straightforward and successful.

Assurance of the ideal machining parameters.

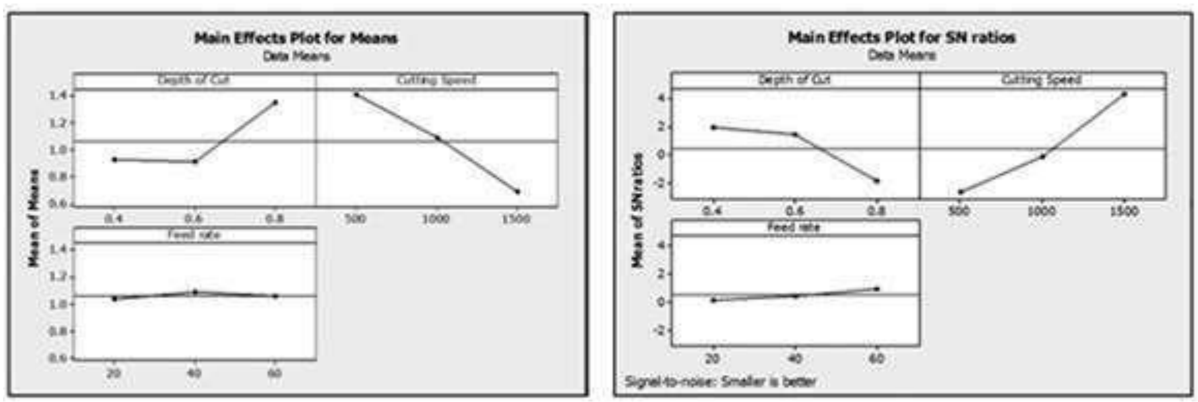


Fig. 4.3 Ra means and relative effects S / N for each of the control factors

- **Surface roughness**

The-littler the-better trademark was utilized to decide the littlest surface roughness (R_a) that would be the perfect circumstance for this examination. In the meantime, the bigger S/N proportion would be anticipated as the best reaction given in the machine set-up framework which would be the perfect circumstance. The charts in Fig. 22 are utilized to decide the ideal arrangement of parameters from this exploratory outline.

From the charts, the control factor of the depth of cut (A) at level 1 (0.4 mm) demonstrates the best outcome. Then again, the cutting rate control factor (B) gives the best 46 results at the level 3 (1500 rpm). Then, the feed rate control factor (C) gives the best outcomes at the level 3 (60 mm/min). There are no contentions in deciding the ideal depth of cut, axle speed and feed rate and the criteria of the least reaction and most noteworthy S/N proportion were taken after. Along these lines, the improved blend of levels for all the three control factors from the examination which gives the best surface complete was observed to be A1– B3– C3.

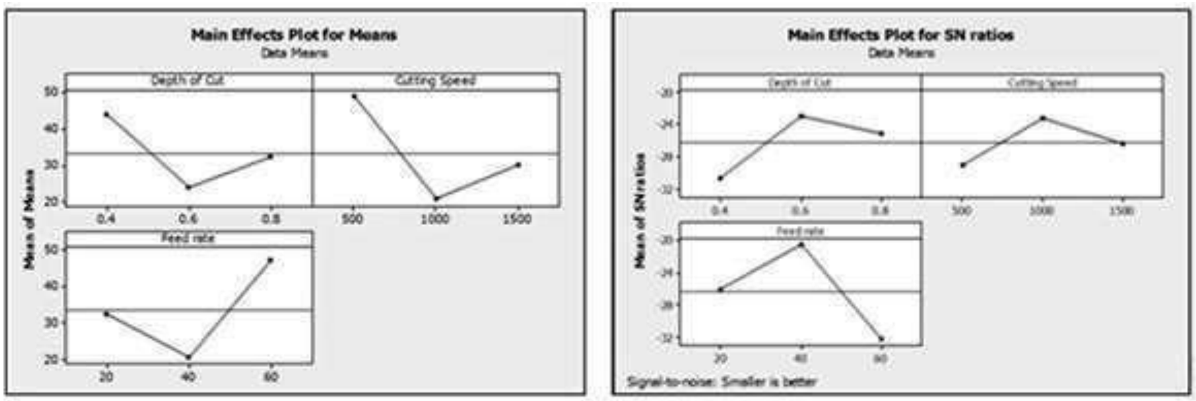


Fig. 4.4 Fr means and S/N ratio effects for each control factors.

- ## Cutting force

In the other reaction factors, the resultant cutting force (Fr), the-littler the-better trademark was utilized and the littlest resultant cutting force esteem would be the perfect circumstance. The control factor of the depth of cut (An) at level 2 (0.6 mm) demonstrated the best outcome. Other than that, the cutting speed control factor (B) gave the best outcome at the level 2 (1000 rpm). Then again, the feed rate control factor (C) demonstrated the best outcomes at the level 2 (40 mm/pm). There were too no contentions occurring in deciding the ideal depth of cut, shaft speed, and feed rate while the criteria of the most minimal reaction and most noteworthy S/N proportion were taken after. Accordingly, the upgraded blend of levels for all the three control factors from the examination which gives the least cutting force was observed to be A2– B2– C2.

4.4 Morphology Study

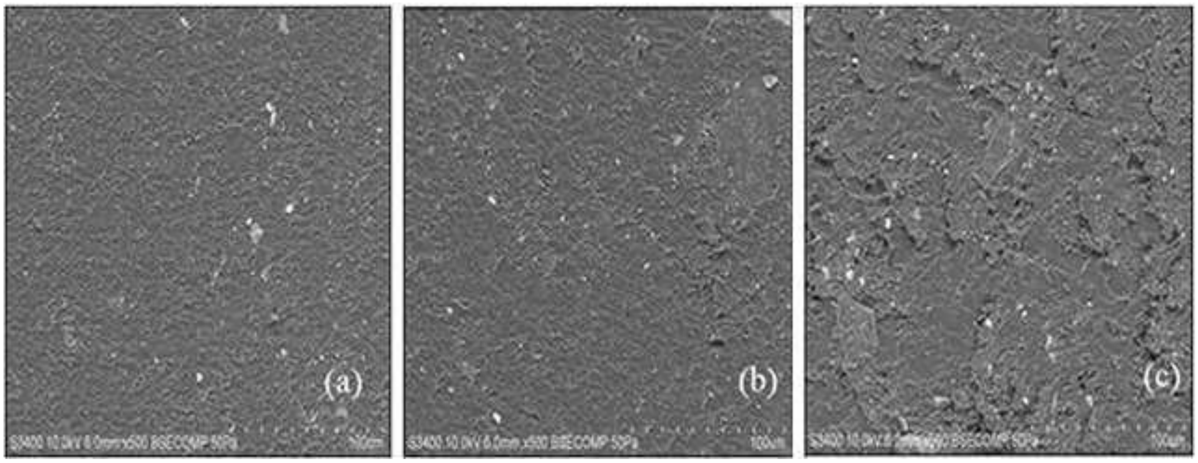


Figure 4.5. SEM study of the surface morphology. (A) Lowest Ra (b) Ra and means (c) the higher Ra

From the examination of surface roughness, there are three ideal blends of control factors which give three unique reactions in surface roughness esteem that can be distinguished. The first control factor blend which gives the most minimal surface roughness esteem is A1–B3-C3. In the interim, the second control factor mix of A2–B2–C2 gave a medium surface roughness esteem (R_a).

For the third mix of the control factors, A3-B1–C1 gives the most astounding surface roughness esteem (R_a). In Fig. 4.5, the surface morphology contemplates were done on all three ideal surfaces. From the analysis, the surface morphology pictures as far as roughness for each of the three machining lines can be believed to differ following the variety of surface roughness esteem.

4.5 Material Removal Rate (MRR)

The material evacuation rate was acquired by a 3x3 exhibit by keeping axle speed consistent. The levels appear in table 4.4.

Table 4.4. The parameters for an orthogonal array

LEVEL	DEPTH OF CUT	FEED RATE
1	0.4	20
2	0.6	40
3	0.8	60

The outcome got for MRR can be found in table 4.5 by the increase of depth of cut and feed rate.

Table 4.5. Orthogonal array

Exp.No	DEPTH OF THE CUT	FEED RATE	MRR
1	0.4	20	48
2	0.4	40	96
3	0.4	60	144
4	0.6	20	72
5	0.6	40	144
6	0.6	60	216
7	0.8	20	96
8	0.8	40	192
9	0.8	60	288

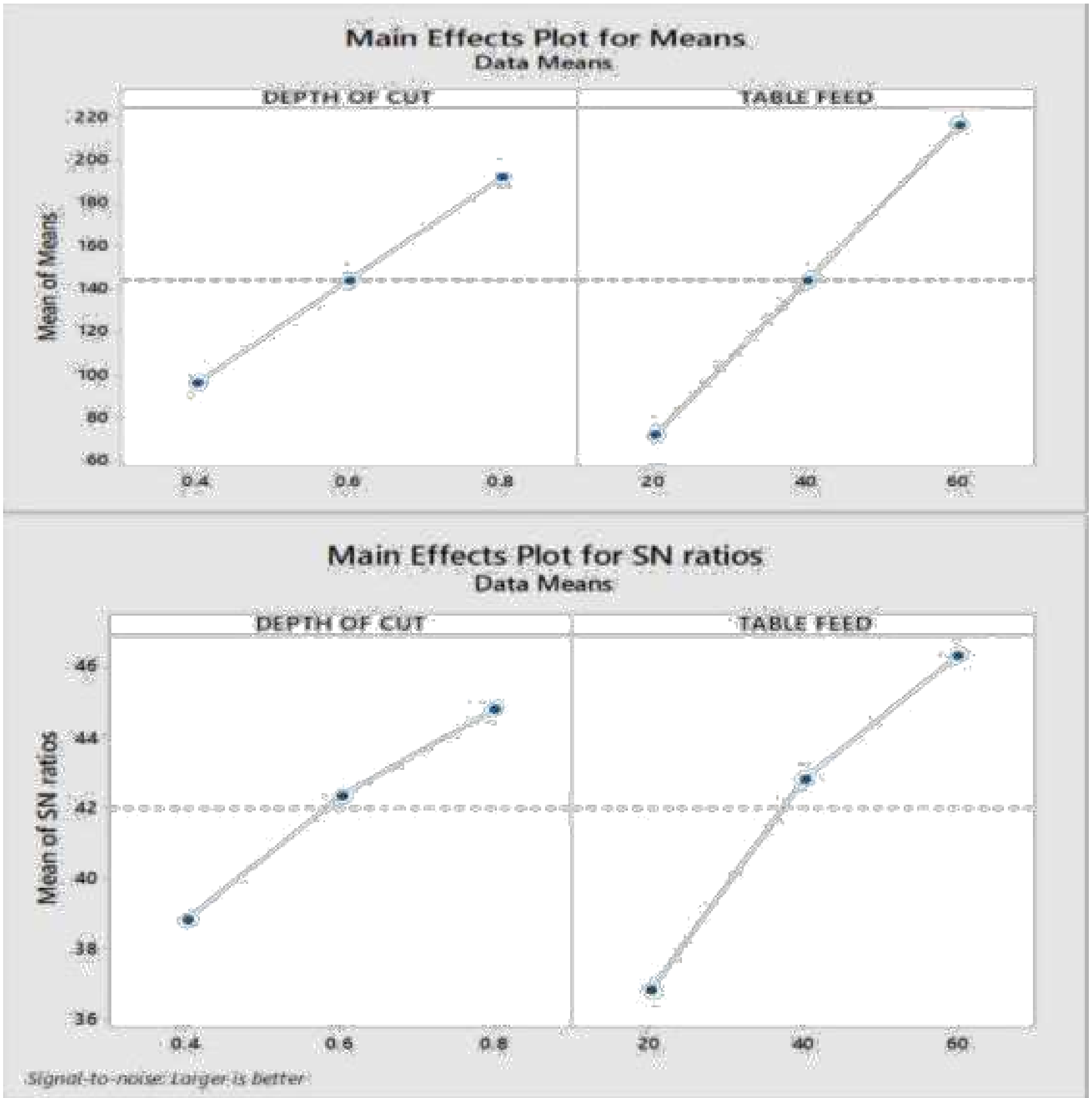


Fig. 4.6 MRR means and S/N ratio effect for the various factor

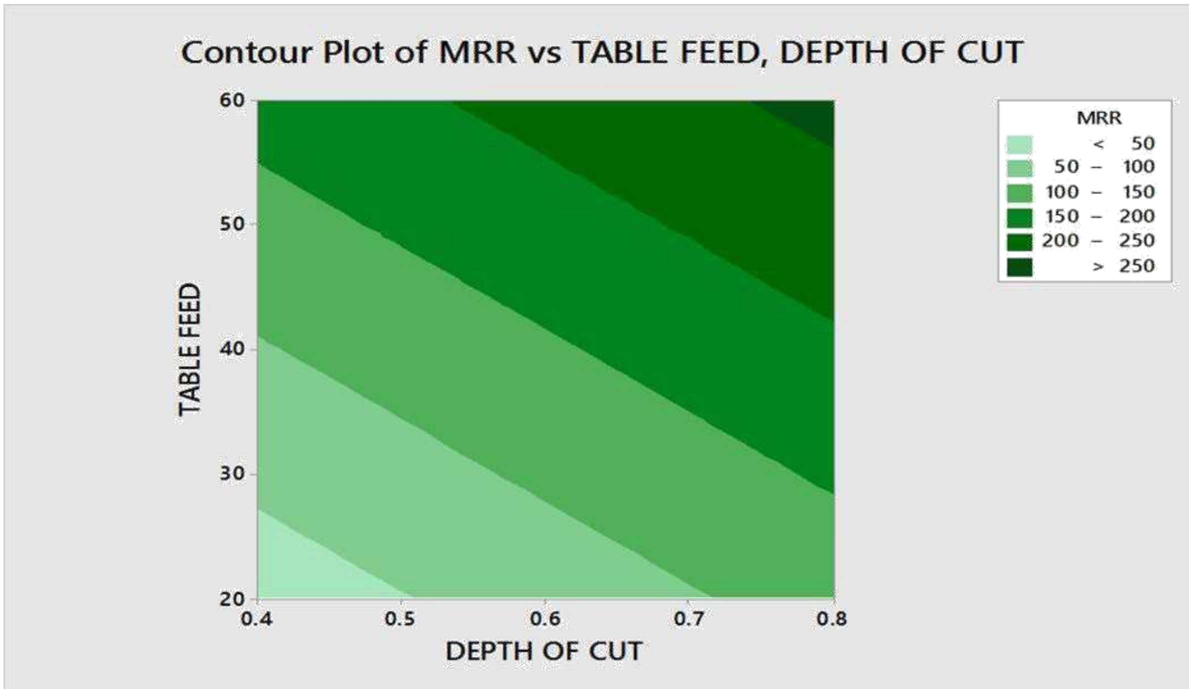


Fig.4.7: Contour plot of MRR vs Table feed, depth of cut

Analysis of Variance (ANOVA)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	2	44928	22464.0	58.50	0.000
DEPTH OF CUT	1	13824	13824.0	36.00	0.001
TABLE FEED	1	31104	31104.0	81.00	0.000
Error	6	2304	384.0		
Total	8	47232			

Contributing Factors Percentage of Individual

The depth of cut---- 30.769 %

Table feed -----69.230 %

Regression Equation Using Anova:

MRR = -144.0 + 240.0 DEPTH OF CUT + 3.600 TABLE FEED

Chapter 5

Conclusions

Graphene oxide nanocomposite has been effectively synthesized at enhanced conditions. FTIR and XRD plainly uncover the successful formation of GO. Machining i.e milling was finished effortlessly on the nanocomposite and Taguchi examination was performed to check the ideal cutting parameters from various levels and mixes. In the wake of getting all the outcome, we would now be able to infer that Taguchi examination is a productive method to decide the ideal mix for the most reduced surface finish, material removal rate and cutting force. Furthermore, the small scale structure surface morphology contemplate was introduced on the visual variety of machined surface roughnesswhich is by all accounts indistinguishable from the variety of surface roughness esteem.

Chapter 6

Future Scope

There is a wide scope for future investigators to explore many other aspects of current research work. Some recommendations for future research include: -

- Study of machining behavior of functionalized composite by doing functionalization by amide group.
- Study of response of composite in study to other aspects of machining.
- Nano composite may be combined with hybrid composite to arrive at variety of new results.
- For use in industrial mass production, after analyzing their cost effects according to their application.

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