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Mechanical Behavior and surface modification of CNTs that are functionalized with Polymer Composites

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Abstract: In the present investigation, the aim was to improve the modification of the surface of the CNTs with different chemical solutions and the characteristics of the F-CNTs reinforced with polymeric composites, so as to promote uniform dispersion and improve the bond between CNTs and polymers. To this end, the CNTs were initially treated with various methods such as acid treatment, silane treatment, thiolation treatment and amine treatment. The functionalization CNTs were characterized by the Fourier transform infrared (FTIR). The main objective of this work was to improve the mechanical characteristics of a polymer composite using nano-reinforcements. The superior properties of nanotubes (CNT) make polymeric compounds a potentially reinforced agent. The interfacial adhesion between the CNTs and the uniform dispersion of the CNTs and the polymer are critical factors that need to be improved to improve the mechanical properties of the polymer compounds. The effect of F-CNT with the polymer content in the composite on the flexural strength, flexural modulus, tensile modulus, tensile strength and fatigue life is assessed.

Keywords: Functionalized-CNT's, FTIR, tensile, fatigue and flexural.

I. INTRODUCTION

Since Iijima's "Breakthrough and Milestone" article of the year (1991) [1], the focal point of carbon nanotubes (CNT) has been an in-depth investigation thanks to their fantastic mechanical, electromagnetic and composite performance. CNT applications have recently been recorded, for example, in Nano devices, field emanation shows, hydrogen storage, moment support, filter test microscopy tests and Nano devices, nano / biosensors, etc. Interesting nuclear structure, high viewing ratio and remarkable mechanical characteristics (quality and adaptability) make CNT perfect fortifying fillers, for example in the year (2007) Shen.J [2] observed the double increase in quality of flexion including 1% by weight of MWCNT without amine Critical impact on the flexural modulus. Comparative results were performed by Chen [3] for epoxy resin nanotubes in epoxy compounds. Furthermore, Zhang [4] demonstrated that a low content of MWCNT improved its dispersion on an epoxy grid and mechanical properties, but a higher MWCNT substance could cause the disintegration of mechanical properties. CNT compounds also show significant improvements in electrical conductivity. The electrical and mechanical properties of CNT polymer compounds appear to be influenced by different nanotube cleaning states or potential adaptation. Tamburri [5] saw that the conductivity of poly-1, 8-diaminonphthalene was charged with SWCNT increments by extending the CNT element due to the proximity of the –OH and –COOH groups presented by the mixed treatment in the untreated application. < HNO_3 < KOH < HNO_3 / H_2SO_4. Kim and colleagues [6] observed that the "conductivity" of the epoxy / MWCNT compounds decreases with the increase of the oxidation time and the temperature of MWCNT in HNO3. John Kathi et al [7] organized rubber resin nano composites and distinctive weight percentages of unmodified, silanized and oxidized multiple-walled carbon nanotubes (MWCNT) according to the mold formation strategy. Analyze the bending properties of the MWCNT content with impact. The results assumed that the expanded and improved flexible modules stack and MWCNT resistance were observed. The mechanical performance characteristics decreased when the MWCNT content exceeded 0.2% by weight due to the assembly of MWCNT. Therefore, the results demonstrate the impact of functionalization on the bond between the bonded edge between the resin and the MWCNT.

In this work, it controls the adequate dispersion of CNT in the polymer through functionalization, silanization, treatment with amines, etc. It was learned that by modeling the adhesions between CNT and the polymer due to the low compaction between them. To destroy these bonds between CNTs and the polymer, it was necessary to simulate the bonds of the substance in the walls of the CNTs to obtain a strong bond with the polymer. Modern advances in the field of nanotechnology require the improvement of nano composites to improve mechanical properties. Further improvement of CNT dispersion in polymers through functionalization and silanization prior to dispersion in the polymer.

II. EXPERIMENTAL MATERIALS

Acetal (purchased from the Korean brand K700) is used to work with a thermoplastic polymer. MWCNT (purchased from SKYSPRING NANOMATERIALS, USA) are used in this work. Chemical products used: hydrochloric acid (HCl), sulfuric acid, nitric acid, ethanol and distilled water (obtained from LOTUS ENTERPRISES), thionyl chloride, acetone (from COASTAL ENTERPRISES).

A. Purification of Multi Walled Carbon Nanotubes (MWCNTs)

First, calculate the raw MWCNT which is calculated to remove any impurities present. Take untreated MWCNT for the calcination treatment Fig. 1 (a) at 545 ° C for 45 minutes. Concentrated hydrochloric acid was treated with calcined MWCNT to remove MWCNT impurities using a consolidated method. Take 500 ml of HCl and 500 ml of distilled water in 10 g of MWCNT, which is placed in a round-bottomed flask. Using a magnetic stirrer, mix the solution for 5 hours. Fig. 1 (b), after which the impurities float on the surface of the solution. They are removed, obtaining a pH value of 6 at paper pH.

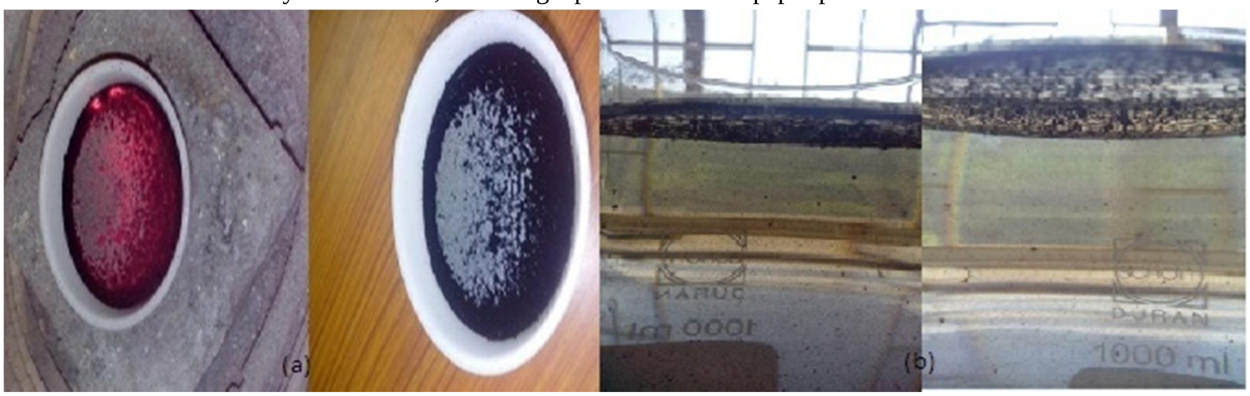

Figure1: (a) After Calcinations Impurities formed on the Top (b) Impurities Floating on the Top

B. The Process of Oxidizing MWCNT's

After purification with hydrochloric acid (Hcl), the solution is oxidized with 1200 ml of a 1: 3 mixture of 300 ml of H_2SO_4 acid, 116 ml of HNO_3 and 784 ml of distilled water in a circular bottom flask with a condenser for 8 hours at 110 ° C Fig. 2(a), distilled water is used to remove any balanced acid solution from the surface of the nanotubes. After the impurities are about to float on the solution. These are eliminated by obtaining the pH of 6 on pH papers. Washing with water must be carried out at least 3 or 4 times for the acid residue to become basic. This means that the solution becomes neutral, indicating that the solution is free of all types of impurities.

Subsequently, these treated MWCNTs are filtered with a series of filters with a 0.22 µ filter paper Fig 2(b), then this purified MWCNTS. Until a pH value of 6 was obtained, MWCNT was required to remove the acid solution with distilled water.

Figure 2: (a) Refluxing Setup (b) Filtration Setup

After the straw collects the MWCNT in the crucible to remove the water content in the nanotubes and places the crucible for 8 hours in the vacuum chamber to dry the MWCNT, as shown in Figure 3, after which the MWCNTs are similar to solid crystals. and are powered by small particles [8-10]. This example is presented for FT-IR analysis to verify the connection to MWCNT. After filtration, the CNTs were collected in a crucible and placed under vacuum to remove moisture, after which they were ground and the sample was introduced into an IR oven to control oxidation.

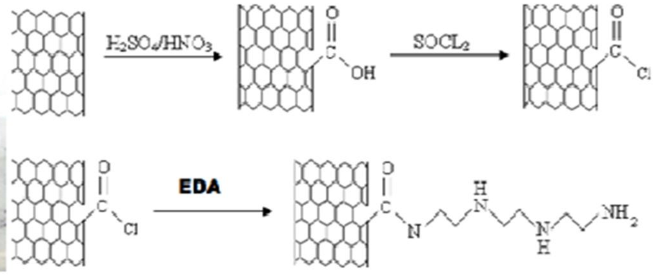

Figure 3: CNT's collected during filtration, Figure 4: chemical functionalization's of CNT's

C. Preparation of functionalized MWCNTS

In this process 1.8gms of MWCNTs are dispersed in 2% of silane solution. Mix both solution like 5% of distilled water and 95% of ethanol. After preparation of that solution, it was stirred for 5hrs by maintaining 700C temperature trough out the process. To neutralize the solution, wash with distilled water to get Ph value of 6. Filter the collected MWCNT's and remove the moisture content with vacuum process for 7hrs. After that MWCNTs are like solid crystals and this are powered to fine particles and sent to FT-IR Analysis. Finally when compared to the oxidation of MWCNT's, shown in Fig. 4 the silanization**[7,12]** have the better values when compared in the FT- IR analysis.

Similarly in the same way the functionalization process is carried for thionalation and amine treatments also. In which the oxidized cnts are taken then processed further with different chemicals depending upon the treatment type. In thionalation process the oxidized CNT's are treated with thionyl chloride and refluxed for 24hrs. It is added as 20ml per 1gm of CNT's. The rest of filtration and drying process is as mentioned for previous processes. Later given for FTIR analysis.

Amine treatment begins with taking thionalated CNT's from above process as input. They are treated with EDA (Ethyelene di-amine) and refluxed for 24hrs.Then the rest of process is same as above, finally given for FTIR analysis for checking bond formation in the material.

III. RESULTS AND DISCUSSIONS

A. Fourier Transform Infrared Analysis

In infrared spectroscopy, of FT-IR, IR radiation was passed through a collected sample, so that it absorbed some of infrared radiation and some radiation was passed through (transmitted) the sample. Various functionalization's on MWCNT have been characterized through FTIR spectra as shown in the below figure. Fig. 5(b) shows peaks at 3400cm^{-1} (Stretching frequency of–OH) and 1380 – 1740cm^{-1} region which can be attributed to C=O present due to oxidation by acid treatment.

When we treated Socl2 (thionyl chloride) with acid treated CNT we observed an additional prominent peak at 610cm^{-1} which can be attributed to C-Cl bond vibration which indicating the formation of OH-C-Cl moiety. Fig. 5(d) is FTIR spectra for the sample treated with EDA after thionilated CNT fig(c). The peak at 2838 and 2923cm^{-1} due to stretching frequency of –CH group present in EDA.

A small hump at 1035cm^{-1} and 3450cm^{-1} be attributed to –NH bond which arises because of EDA. Fig 5(e) is the FTIR spectra of sample treated with APTS after acid treatment of MWCNT. The band at 1021cm^{-1} can be attributed to Si-O-Si vibration match to siloxane units which is formed during silanization period.

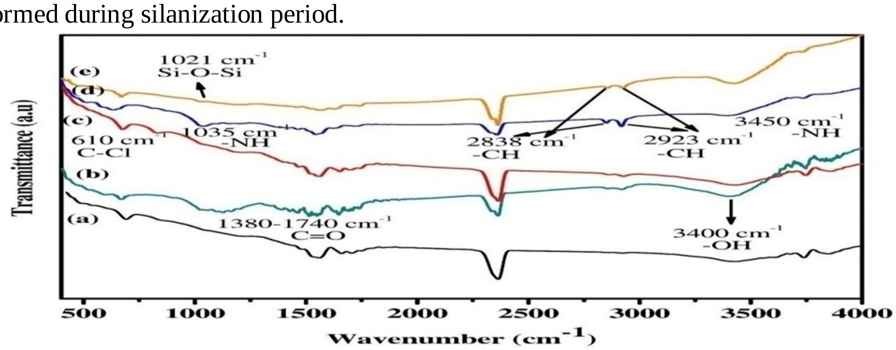

Figure 5: Comparison on functionalization's

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B. Mechanical Properties

From the below Fig. 8&9 we can say that there is significantly improvement in Tensile properties of acetal after mixing with functionalized CNT's. In particular when compared within the functionalization's Silane treatment has shown much better performance when compared to other functionalization types **[11-15]**. Next to that Amine and Thionilated CNT functionalization's have shown better results and stood next to Silane treatment.

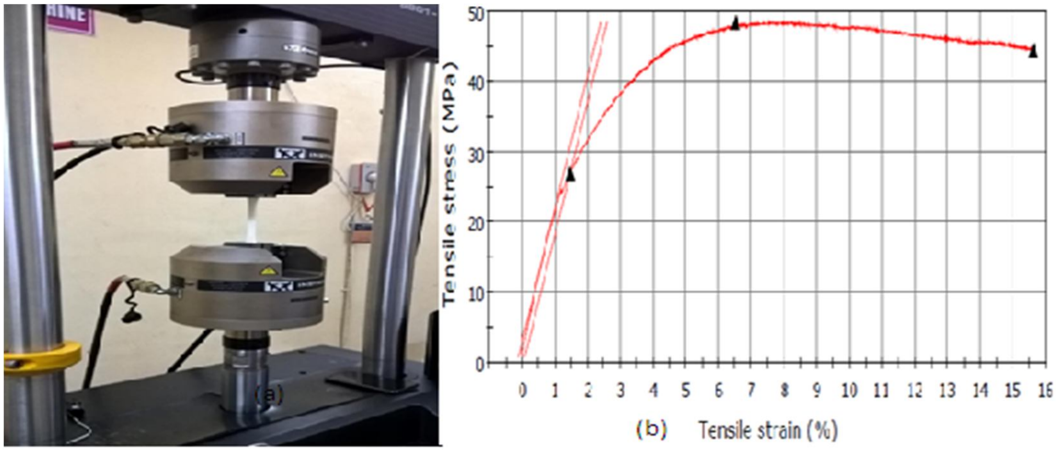

Figure 6: (a) Tensile testing of specimen (b) stress – strain graph of pure acetal 1.Proportional limit 2. Ultimate tensile strength 3. Breaking

Figure 7: (a) Specimen after tensile test pure acetal (b) Specimen after tensile test pure acetal

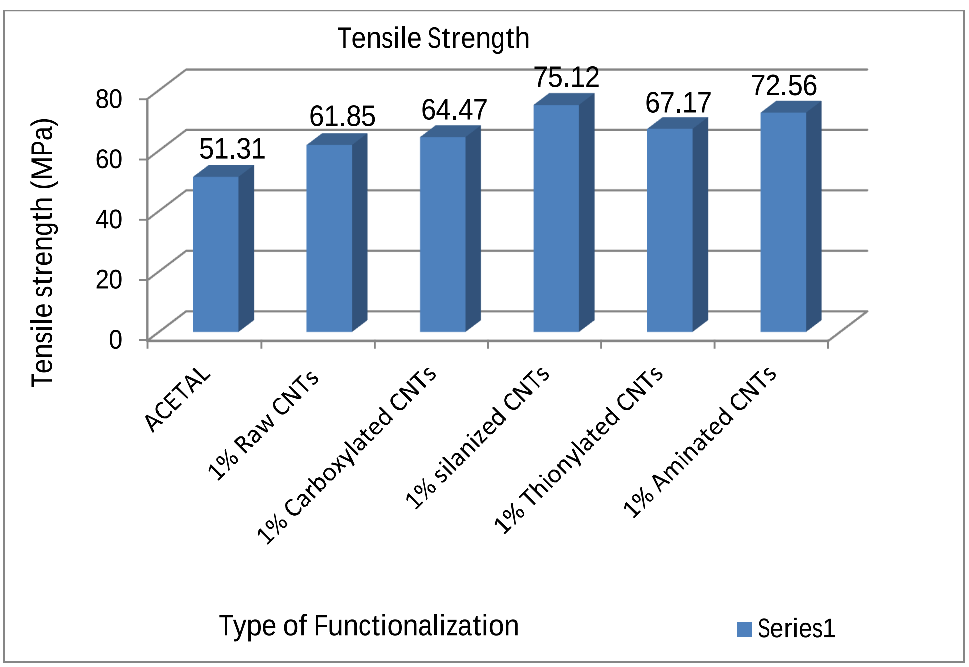

Figure 8: Comparison of Tensile Strengths

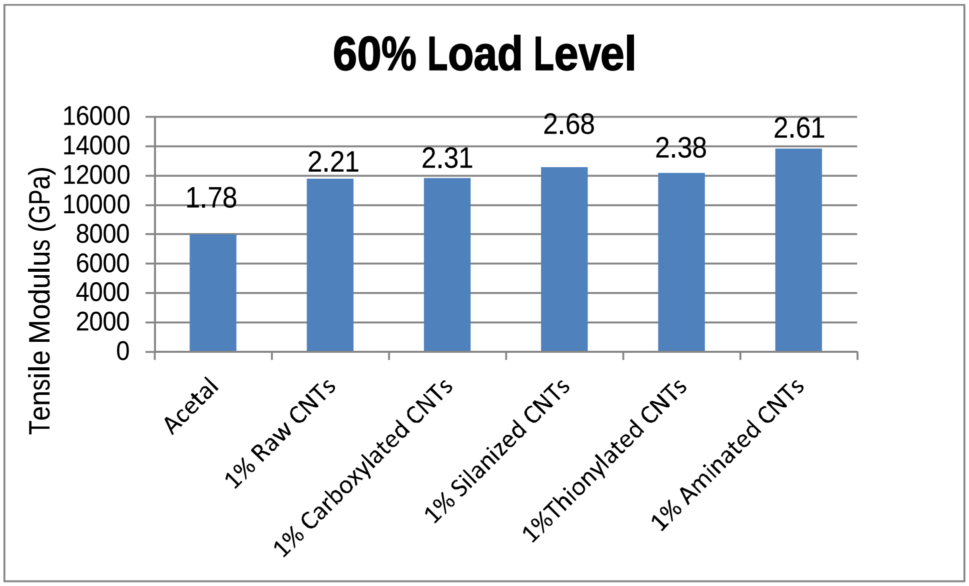

Figure 9: Comparison of Tensile Modulus

C. Fatigue Test

From the below Fig.10 we can say that fatigue properties have been improved in comparison to the base material i.e. pure acetal. By adding functionalized CNT's to the material have resulted in increase of fatigue life of the material. We can clearly see from the graph plotted above that Silane treated CNT's have much impact on the material. Next to that the functionalization's of Amine and Thionylation.

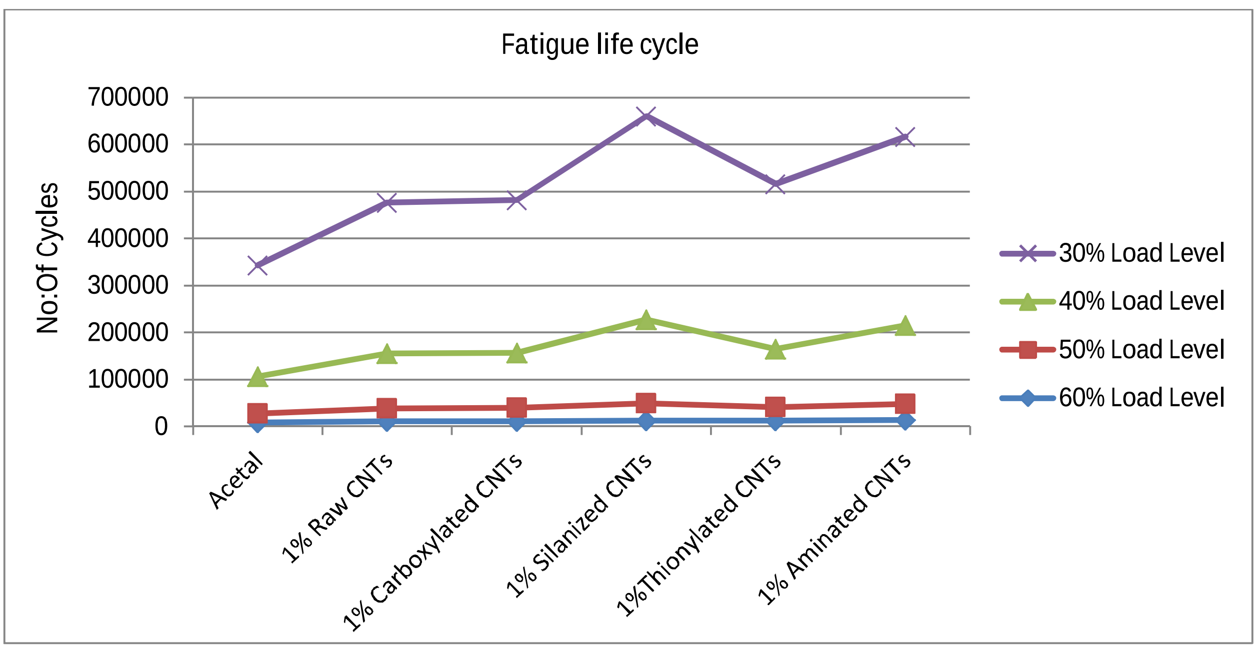

Figure 10: Fatigue life cycle graph

D. Flexural Test

Place the specimen on the three point bending at a specified rate, specimen must lies on a support span and apply the load at the center of the specimen with loading nose. Variables for the experiment is the speeds of the loading, span support and maximum deflection Fig 11(a), these variables are depend upon the thickness of the specimen and ASTM and ISO standards. If consider ASTM D790, the specimen reaches 5% deflection stop the test, otherwise it will break after 5% deflection as shown in fig 11(b). If select ISO 178, after breaking the specimen stop the test. Still the specimen is not fractured, continue the test as long as possible and report that the stress is 3.5%.

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E. *Geometry size of Specimen*

For the experiment results different sizes of specimens can use, but regularly used geometry for ISO and ASTM is 10mm x 4mm x 80mm and 3.2mm x 12.7mm x 125mm (0.125" x 0.5" x 5.0").

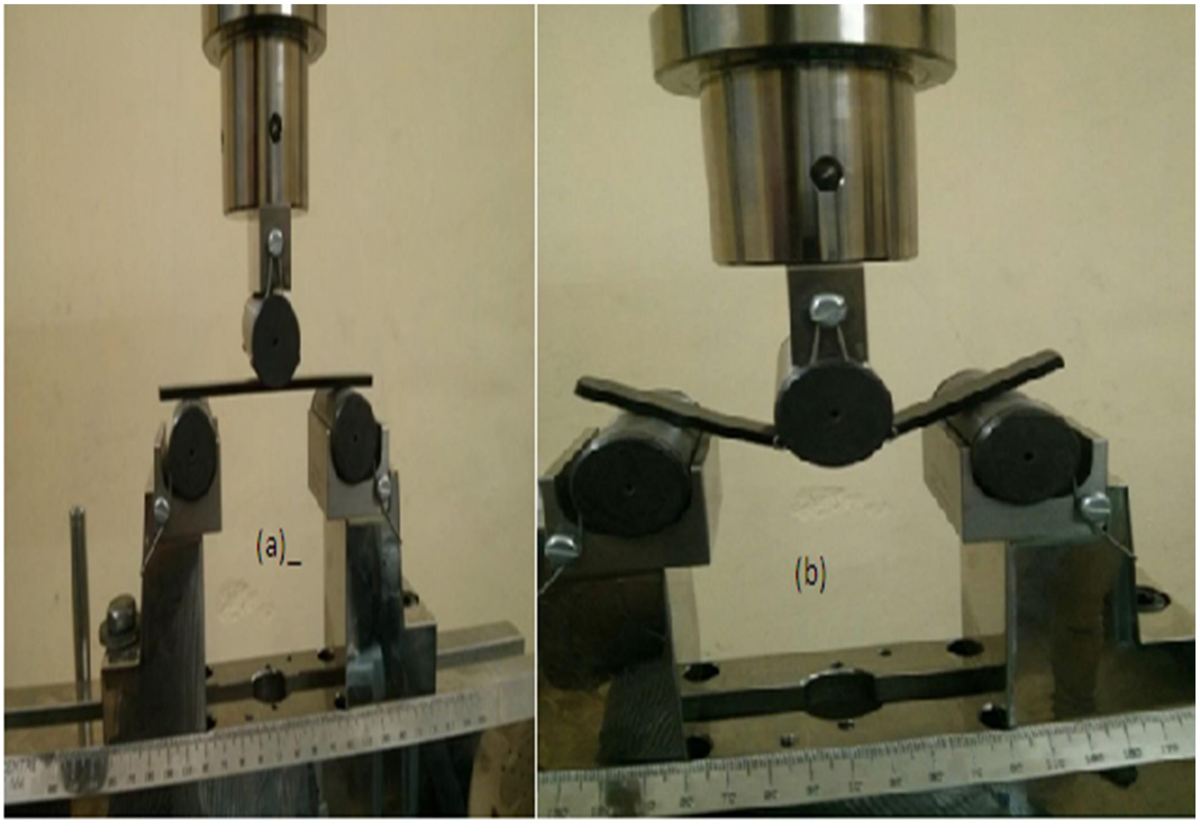

Figure 11: (a) Before commencing test (b) During flexural test

From the below Fig. 12&13 we can say that both the flexural strength and flexural modulus have been improved in comparison to pure acetal. This is because of functionalization of CNT's. Different functionalization's resulted in different percentage in increase of properties of material. From the graph it is clear that Silane treatment has shown better results and next to it is Amine and thionylated functionliztions.

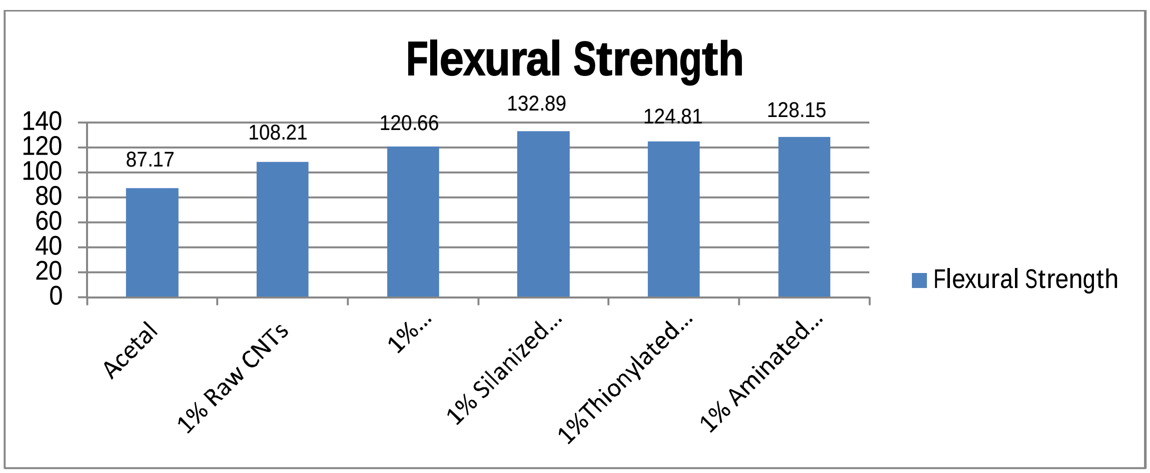

Figure 12: Comparison of Flexural Strength

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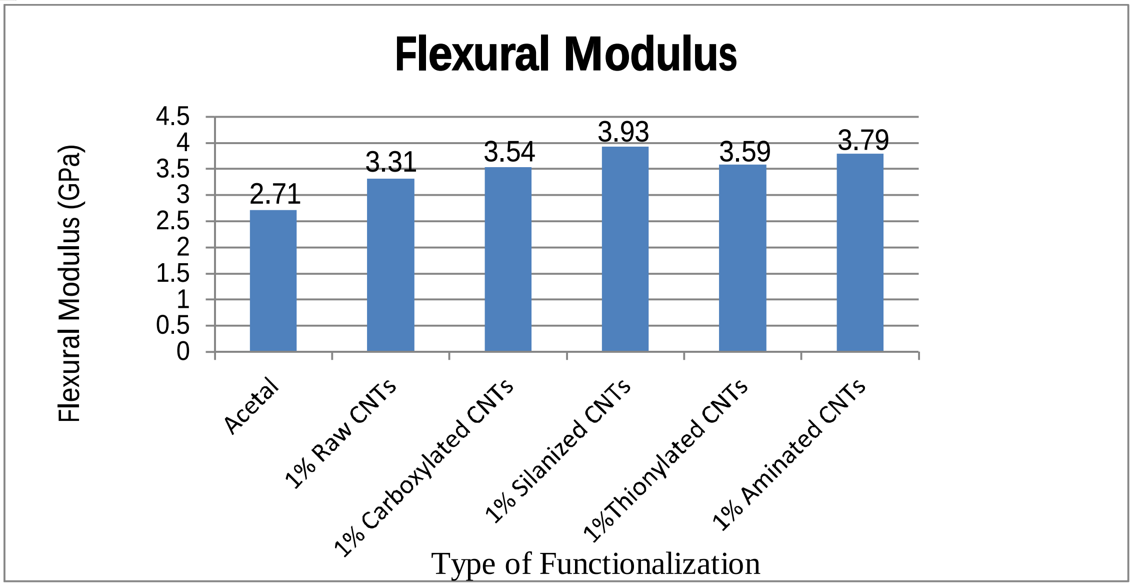

Figure 13: Comparison of Flexural Modulus

IV. CONCLUSION

This is to conclude that the data received from the modification of Surface after optimizing the performance parameters by observing the functionalizations in CNT at 1% by weight, we have achieved the following resolutions. There by diffusion of CNT functionalized in the polymer it gives predominant mechanical properties. MWCNTs treated with silane dispersed in a compound improve the mechanical properties and also show an increase in the ratio of the modulus which is not as indicated by the various compounds. In all cases, compounds treated with amine and thionyl is excluded with great distinction. MWCNTS functionalized with silane and other functionalizations improve the good adhesion between NTC and the polymer matrix by changing the properties of the matrix and henceforth the properties of the compound increments. Thanks to this, it has improved the properties of Flexural and Fatique. As for the tensile properties, the fatigue and flexion of the silanized compound were predominant, followed by the amine, the thionyl and the oxidized compound and, finally, by the unmodified compound.

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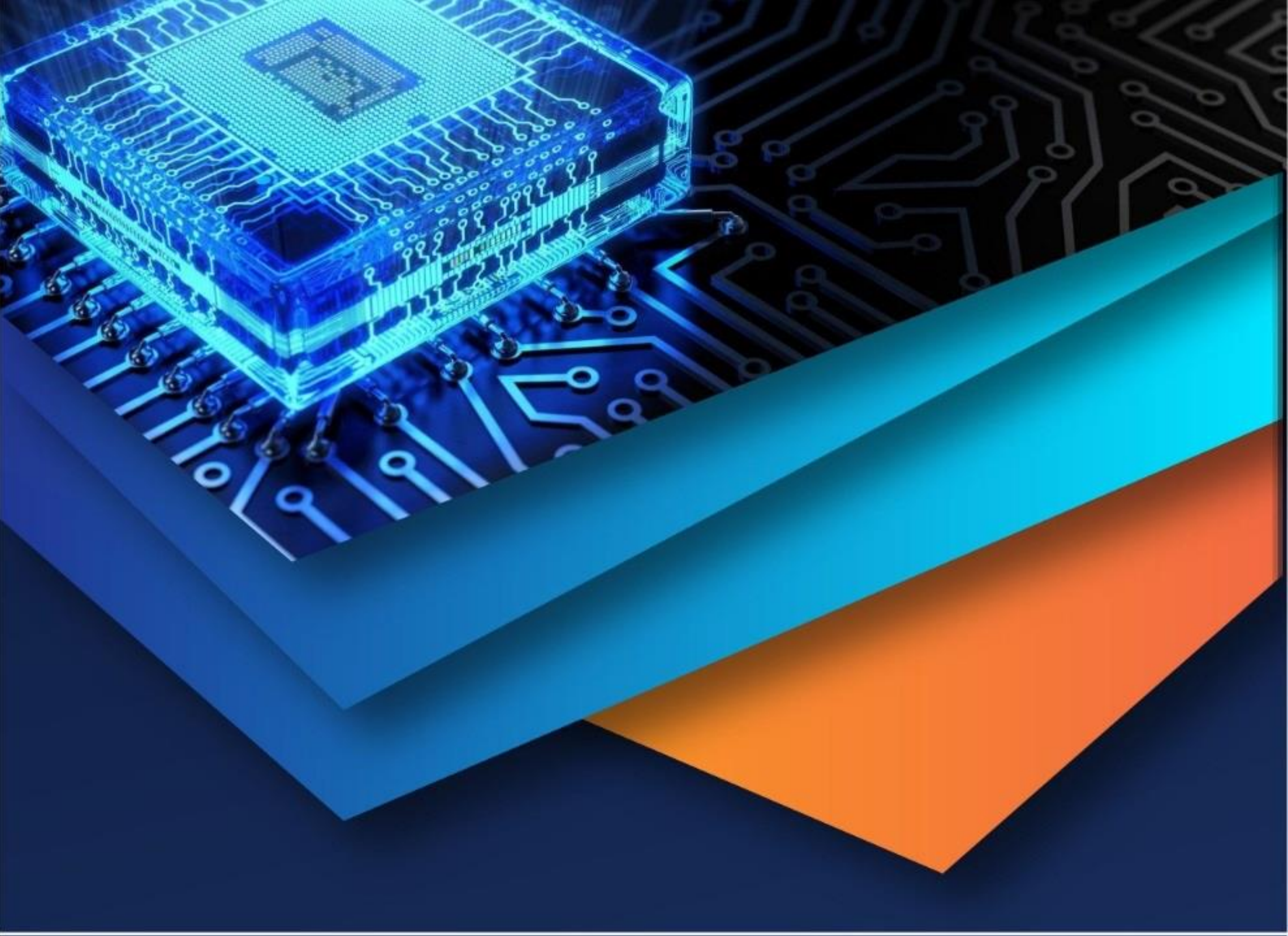

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