Microwave Absorption by Melamine/Carbon Nano Fiber Composite-11

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***Abstract:* - Microwave absorption in the frequency 2-8GHz (S & C band) by carbon nano fiber (CNF) and its composite with melamine has been studied. CNF was synthesized from cotton fiber and activated with a mixture of Ni+Co oxide to study the microwave absorption. Various thicknesses of activated CNF thin film (3-10mm) were studied and it was found that films thicknesses of 3mm and 6mm had the highest flat Reflection Loss of almost 94% and 96% for frequencies 6-8 GHz and 4-6 GHz respectively. Composite of CNF:Melamine (1:5) of thicknesses of 4.5mm and 6 mm showed the best flat reflection loss of 99.88% for frequencies 6-8 GHz. Melamine polymer of 3mm thickness showed less than 5% Reflection loss in the frequencies 6-8 GHz. The results of microwave absorption observed with CNF films were found to follow the theoretically expected values. Activated CNF films were characterized by SEM, XRD and Raman spectroscopy**

***Keywords* – Carbon Nano Fiber, Microwave Absorption, Melamine-CNF composite**

I. INTRODUCTION

Rapid development in the field of communication has attracted many scientists around the world in areas related to microwaves. Microwave absorbing materials have profound defense application such as to bypass the tracking of an aircraft or submarine on radar (or SONAR) systems. The operation of electronic devices onboard are affected due to electromagnetic interference caused by communication devices. Such devices are not only limited to radars, but also to mobile phones, electronic circuits, Laptop etc. To avoid the interference of electromagnetic waves with the instruments, there is a need for good microwave absorbing materials. Carbon nanotubes, carbon nanospheres are promising candidate for microwave absorption in the range of 4-18 GHz[1-7]. Incorporation of nickel and Cobalt oxide into CNF improve the microwave absorption in the 2-8GHz [4]

Under the present study CNF from cotton fibers was used to make a polymer composite with melamine to study the microwave absorption in the range of 4-8 GHz.

II. EXPERIMENTAL METHOD

2.1 Preparation of composite

CNF synthesized from pyrolysis of cotton and chemically activated by nickel and cobalt has been used to study the microwave absorption. The method of synthesis of CNF and its activation has been discussed in our previous publication [4]. Activation was done with 5% Ni+Co oxide prepared from their nitrate solutions. A film of activated CNF coated on a copper sheet is shown in Figure1A. The activated carbon was impregnated / suspended in the Melamine resin to prepare the composite. Melamine (4.66g) and Formaldehyde (15 cc) was mixed which gave a composition of 1:5 [8-10]. The two materials were then mixed to form a milky white solution. The mixture was heated to 66^{o}C with continuous stirring for 15 minutes till the solution became clear. Since solution was acidic (pH 5) it was made basic by addition of 1 N NaOH (100µL) to make pH 12. To this solution 1 gram of carbon fiber (10% by weight) was added and solution was stirred to make a homogeneous mixture. This solution was then poured into the mold and allowed to dry slowly which resulted into composite thin film of CNF mixed with melamine polymer (Figure 1B).

The solution was made basic because N-C bond is more stable in basic medium than the acidic medium. Also, NaOH acts as catalyst to facilitate the cross linking of molecules [3]. Melamine is a thermosetting plastic polymer. Since thermoset materials after hardening cannot be melted to form a desirable shape, generally, the liquid is put into the mould before curing.

Figure 1 A photograph of (A) CNF pasted on a copper sheet (B) a paste of CNF with Melamine polymer on copper sheet

2.2 Microwave measurement:

In order to have an idea of loss of microwave power, the reflected power by material using a microwave test bench were measured. Details of the set up of microwave measurements are discussed elsewhere (Nallin *et. al* [4]). Terminating plane surface of the waveguide was cleaned by using polish paper and acetone. The surface was cleaned with a polish paper of 220 grains followed by fine grain paper of 1000 grains. Acetone was used to wipe off the moisture. Then, the surface was fixed to the waveguide using bolts and nuts (Figure 2). The wave source meter and power meter were powered. The wave source meter produces microwaves while the power meter measures the power reflected by the surface (or material). The range (the frequency) is adjustable using the knobs on the wave meter for coarse and fine tuning. The waveguide is coupled to a directional coupler which captures the reflected waves by surface of the material. The directional coupler terminates using a thermistor which sends power to the power meter. For a range of frequencies, their corresponding power reflection was recorded. Clean copper film was used as reflecting and terminating surface.

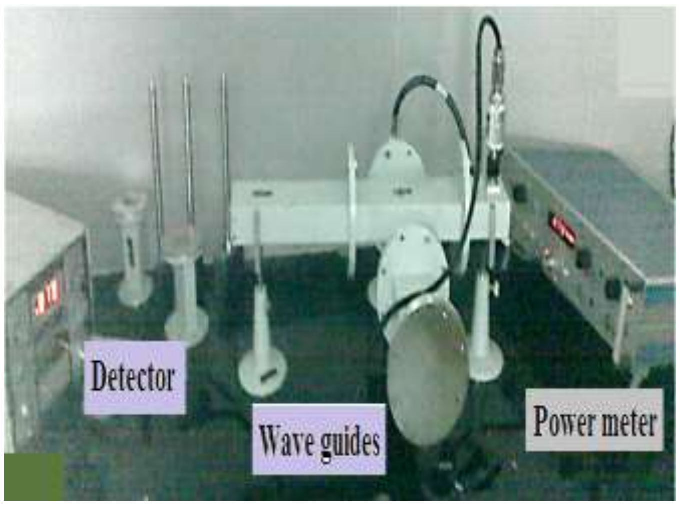


Figure 2 Photograph of microwave set up. Each unit of the setup is labelled.

A reflecting surface (aluminum) was kept above Copper metallic reflecting surface as a reference to measure the microwave absorption. A layer of CNF of specific thickness was prepared (Figure 1A) . Power absorption by CNF was measured by measuring the reflection by reference metallic surface with and without carbon sample. The difference in power between reflecting metal and reflecting Copper metal with CNF was calculated to get the power absorbed by CNF. Similarly the microwave absorption was measured for only film of melamine polymer. Finally by similar procedure % reflection loss was measured with CNF-melamine composite.

The absorption was calculated in percentage by using the following expression

$$Reflection\ Loss\ (in\ \%) = 100 - 100\left(\frac{Reflection\ by\ Sample}{Reflection\ by\ Blank}\right)$$

III. RESULTS AND DISCUSSION

3.1 Morphology

SEM micrograph of the activated CNF shows its diameter 5-8μm. CNF also contains some broken fibers (shown by yellow arrow). Metallic particle present over the surface of CNF fibers are also seen in the micrograph (red arrow Figure 3A).

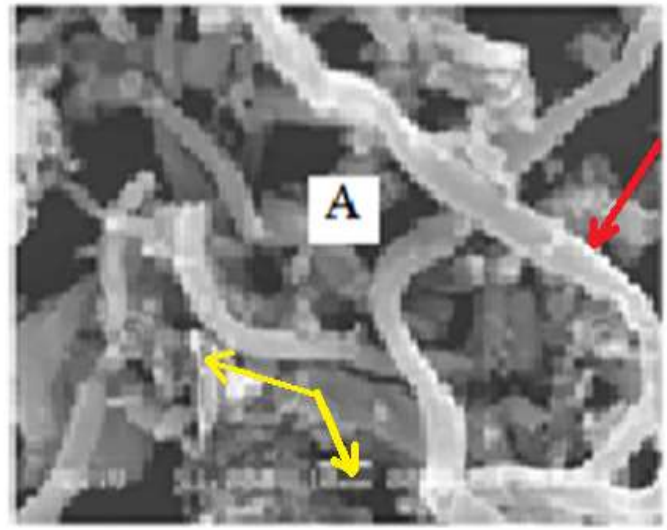


Figure 3 SEM micrographs of activated CNF(A) showing many metallic particles over the CNF (red arrow) and some broken hollow fibers (yellow arrow)

.XRD of activated CNF is shown figure 4. XRD spectra shows the presence of graphitic peak around 2θ = 26 and other peaks are not well developed but are perhaps due to oxides of Ni and Co.

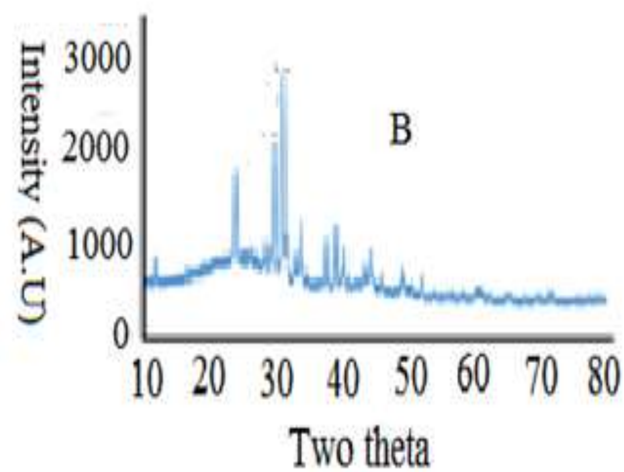


Figure 4 XRD spectra of activated CNF with a mixture of Ni+ Co oxide.

Raman spectra of activated CNF (Figure 5) shows the D peak at 1339cm^{-1} and G-peaks at 1593 and D^{II} peak at 2994cm^{-1}. These peaks suggest that carbon fiber contains some defect (1339cm^{-1}) along with the graphitic (1593 cm^{-1}) nature. The origin of peak at 2994 cm^{-1} not yet fully established but it considered as double resonance peak of G band.

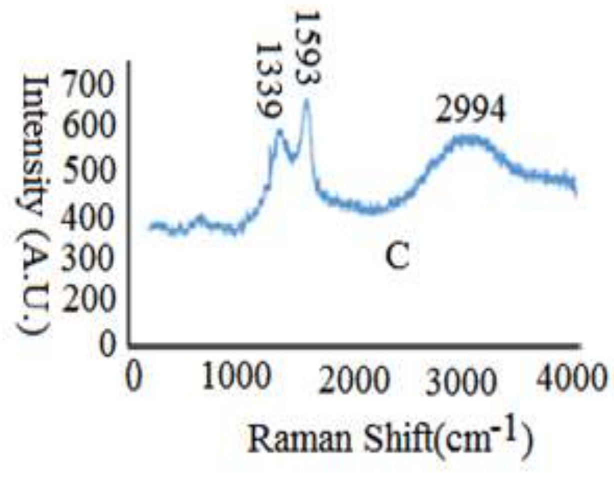


Figure5 Raman spectra of activated CNF wit a mixture of Ni + Co oxide showing specific peak of D-band at 1339cm^{-1} and that G-band at 1593cm^{-1}. The peak at 2994cm^{-1} is not yet fully understood but is considered as double resonance peak of G-band.

3.2 Microwave Absorption

CNF is studied for its microwave absorption property in the frequency range 2-8 GHz wherein the sample was exposed to microwave radiations in the mentioned frequency band. The sample was exposed to microwave radiations in the frequency range of 2 – 8 GHz. Reflection Loss was plotted for the selected frequency band. Measurements of reflection loss were made with three types of samples: (i) Melamine resin alone stacked over a reference copper metal plate of thickness 3mm (ii) CNF thin film of different thickness (3-10 mm) (Figure 1A) and , (iii) Composite of CNF with melamine-(figure 1B) of different thickness (3-6mm) . With some samples the Reflection loss (%) was more than reference samples and reading contained many noises and they were omitted.Hence readings were taken for those frequencies which gave absorption more than reference and contained no noise. Several measurements were taken for each sample. One typical % reflection loss of microwave obtained with these two sets of samples are shown figure 6.

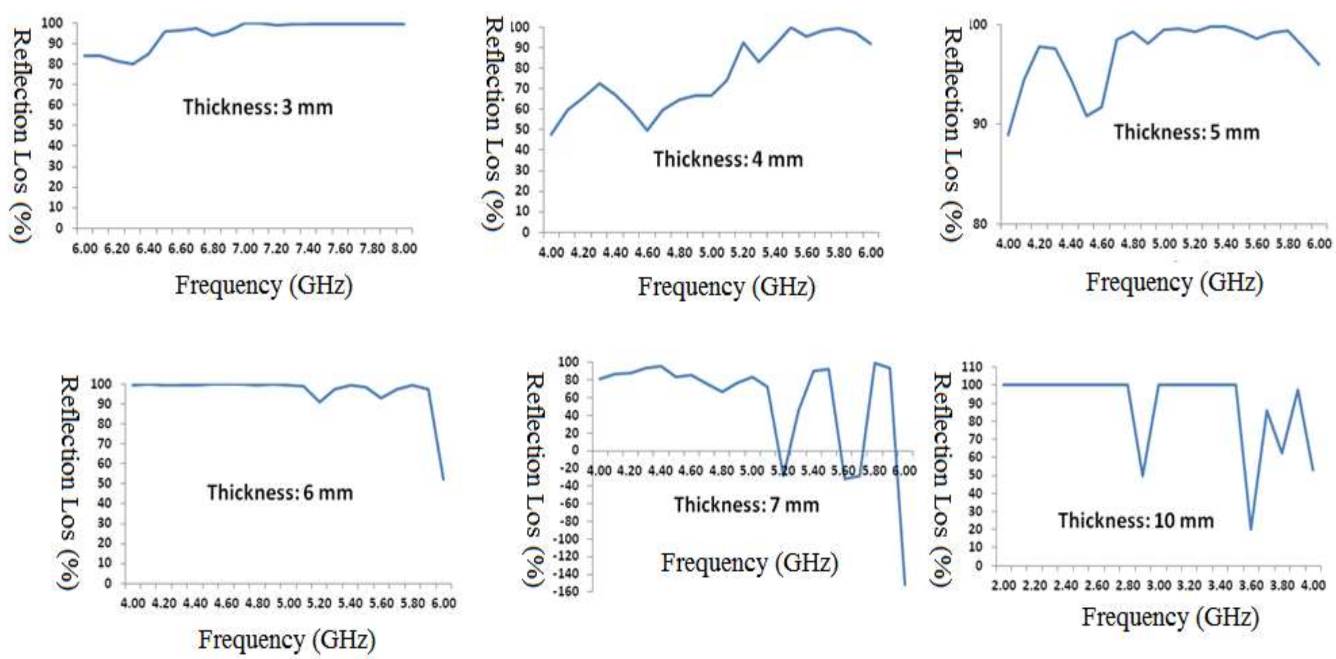


Figure 6. Graphs showing % Reflectance loss for the frequency range 2-8 GHz with thin film of different thickness of CNF.

Examination of the graphs shown in figure 6 suggests that CNF film thickness of 3mm shows flat absorption in the frequency range 6 – 8 GHz while CNF film thickness of 5mm,, and 6mm shows almost flat absorption for the frequency range 4 – 6 GHz. CNF film thickness of 7 mm shows flat absorption for only 4-5 GHz while 10 mm thickness film shows almost flat absorption for 2-3 GHz.

We need to explain these results by taking help of theoretical approach.

Microwaves are normally captured by using an antenna. For resonance, optimum length of antenna should be three-quarter of wavelength ($3\lambda/4$) of the incident wave. At resonance frequency, amplitude of oscillations is more than other frequencies. Since, we are not dealing with antennas; we can try to explain the results with thickness of layer used for absorption.

Required length of antenna for resonance at 6 GHz is calculated as follows:

$$\lambda = \frac{3 \times 10^8\ ms^{-1}}{6 \times 10^9 s^{-1}} = 0.05\ m$$

$$Antenna\ Length = \frac{3\lambda}{4} = \frac{3 \times 0.05 \times 100}{4} cm = 3.75\ cm$$

This suggests that the length of antenna for absorbing 6 GHz should be 3.75cm. In the present work we are dealing with thickness of film rather than antennae. Can the material used for absorbing microwave give similar effect as antenna of thickness of $1/10^{th}$ of the expected length of antenna? If this is expected then the required thickness of CNF for absorbing 6 GHz frequency should be calculated as:

$$Thickness = \frac{Antenna\ Length}{10} = \frac{3.75\ cm}{10} = \frac{3.75 \times 10\ mm}{10} = 3.75\ mm$$

Likewise the thickness of composite for other wavelengths of microwave can be calculated. The values are given in table-1.

Table-1 Maximum thickness (mm) of absorber needed to absorber various microwave frequency (GHz)

Frequency (GHz)	2.2 (D band)	3.3 (D band)	4.0	6.0	8.0
Thickness of absorber (mm)	10.23	6.82	5.63	3.75	2.81

This theoretical calculation suggest that best thickness for absorbing microwave in the range of 6-8 GHz should be between 2.81 and 3.75mm and for frequency 4-6 GHz the thickness should be in the range of 3.75mm and 5.63mm. These conclusions might suggest that irrespective of type of materials used for measuring the reflection loss, the maximum absorption can always be obtained with these calculated thickness. This is not correct. These calculations suggest that we need to find out absorbing material which can absorb these frequencies by using at least these calculated thickness. If a material can absorb same amount of absorption with less thickness, then that material will be more suitable because the total weight of the material will be less.

The experimental results show the absorption of 6-8 GHz is best with film thickness of 3mm while for 4-8 GHz thickness of 5mm and 6mm shows better absorption. Likewise for 4-5 GHz film thickness of 7 mm shows better absorption. Similarly for 2-3 GHz frequencies film thickness of 10 mm shows better absorption. Thus all results of microwave absorption using CNF matches with expected theoretical values (table-1).

In order to get some understanding about the effect of thickness on each frequency range, reflectance loss was calculated from these graphs (Figure 6) for each thickness and for each frequency range. A graph was plotted of reflectance loss for each frequency (in the range of 4-8 GHz) against the thickness of the sample (Figure 7A). In order to compare the results with theoretically expected value a graph was also plotted between expected thickness of the film and corresponding frequency (Figure 7B). The comparison of two graphs suggests that experimentally observed values matches very well with theoretically expected values.

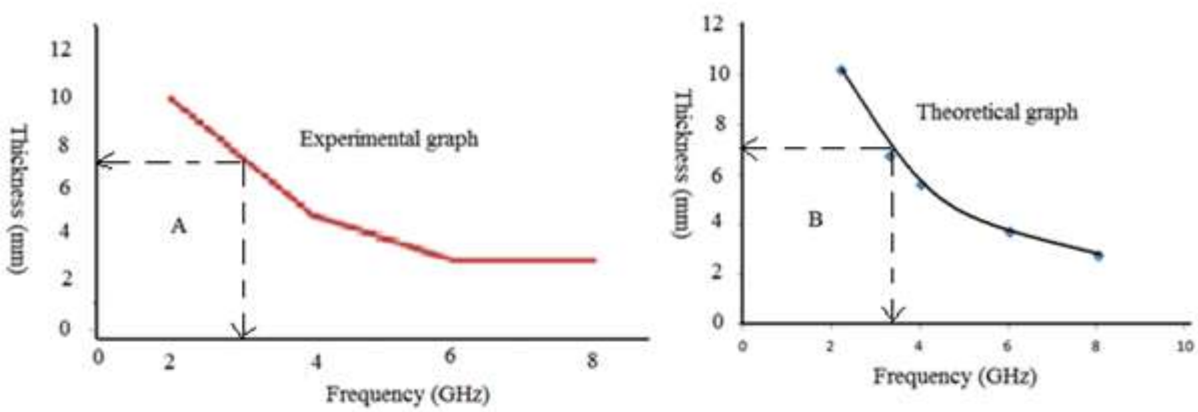
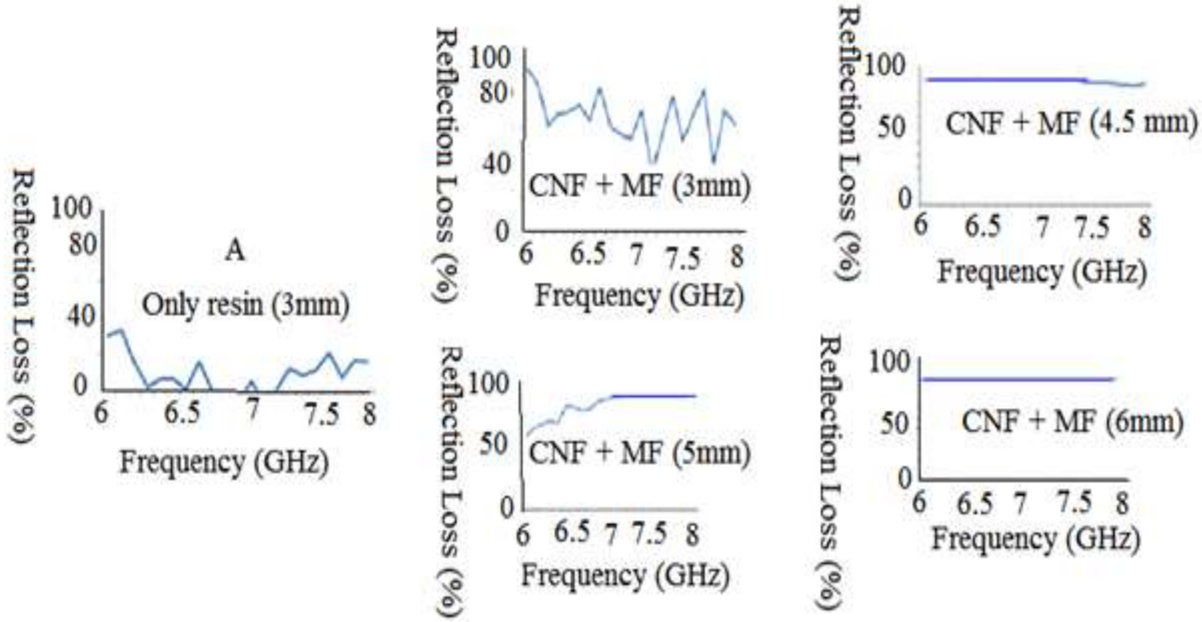


Figure 7 (A) Shows the experimentally observed reflection loss obtained with CNF-composite for various thicknesses for corresponding frequency (B) shows the theoretical values of reflection loss of microwave absorption obtained with different expected thickness for different frequency of microwave. These values are taken from table1.

These experiments were also carried out with films prepared by composite of CNF and Melamin polymer with different thickness keeping ratio of CNF to melamine constant of 1:5. Results of reflection loss (%) obtained with different thickness of film of composite is shown in figure 8. In these measurements Reflection loss (%) obtained with some of thickness of film was less than reference and hence they were discarded.



Figuren8 (A) Percentage Reflection loss obtained with a film of melamine polymer of thickness 3mm and others are the reflection loss obtained with a composite of CNF + melamine polymer for various thicknesses keeping ratio of CNF: melamine 1:5.

The examination of these graphs reveals that like what we observed with pure CNF, composite film thickness of 4.5 mm and 6 mm also show the flat reflection loss for frequency range from 6-8 GHz.

From the results obtained with percentage reflection loss with only CNF or with CNF+ melamine polymer, film thickness of 6 mm shows almost the same behavior with very flat 99% absorption for the entire frequency range of 6-8 GHz. Melamine helps in sticking the powder of CNF together without creating any adverse effect on the percentage reflection loss in 6-8 GHz frequency range.

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