Journal of
Nanoscience and Nanotechnology
Vol. 5, 2117–2120, 2005

Application of Carbon Nanomaterial as a Microwave Absorber

Maheshwar Sharon,[1,*] Debabrata Pradhan,[2] Renju Zacharia,[3] and Vijaya Puri[4]

[1] Nanotechnology Research Lab, Birla College, Kalayan 421304, India
[2] Regional Sophisticated Instrumentation Center, Indian Institute of Technology, Bombay 400076, India
[3] Department of Physical Chemistry, Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, D-14195 Berlin, Germany
[4] Department of Physics, Shivajee University, Kolhapur, India

Microwave absorption (8 GHz to 12 GHz) studies have been made with carbon nanomaterials for the first time. Carbon nanomaterials are synthesized by the pyrolysis of camphor. It is observed that film of carbon prepared under certain synthetic condition, can absorb microwave of either some specific wavelengths e.g., 9.5 GHz and 11.5 GHz or full range from 8–12 GHz to the extent of 20 dB depending upon their preparation condition. Carbon nanobeads seems to absorb the microwave in the range of 8–12 GHz.

Keywords: Microwave Absorption, Camphor CNT, Carbon Nanobeads from Camphor, CNT Microwave Absorption, CVD, Taguchi Analysis.

1. INTRODUCTION

"Microwave" is a descriptive term used to identify electromagnetic waves in the range approximately from 1 GHz to 30 GHz. These waves present several interesting and unusual features not found in other portions of the electromagnetic frequency spectrum. Today microwaves are playing an ever-increasing role in the field of communication. Radar systems are used to locate a target and to determine accurately its position in space with respect to the radar unit. Microwaves are used as a means to determine the distance of the target from the radar unit. Thus making it possible to locate the exact position and velocity of any flying object. But if a flying object absorbs the microwave, it cannot be detected. So electromagnetic wave (EMW) absorbers find diverse application in the present day. In the past, many attempts have been made using different kind of materials i.e., from magnetic ferrites[1–3] to newly advanced polymer based composites[4–5] showing some absorption characteristics. The latest publication[6] on Barium-M-type Ferrite ($BaFe_{12-x}$ ($Ti_{0.5}Mn_{0.5}$) O19) shows the dependence of reflection loss (R.L.) on frequency. The minimum reflection losses obtained from these samples were in the range of −10 dB to −20 dB at the matching frequencies varied from 15.8 to 38 GHz, for samples of $x = 2.5$ to 5 and matching thickness from 0.5 to 3.8 mm. Effective shielding properties of nickel coated carbon fibers[7–8] and carbon composite[9–10] has also been reported. The advantage of using carbon lies in its low weight, corrosion resistance and good conductivity. In addition to this, carbon can be coated on any geometrically complex body by chemical vapor deposition.

Unfortunately, these ferrites absorbers are prone to oxidation. Moreover, these materials need a thick coating to meet practical demands. Therefore, there is need to develop materials which are non-corrosive, stable against corrosion and even thin coating should be sufficient to absorb microwave. Carbon being stable to corrosion and extremely easy to coat on any metal may prove to be a useful absorber for microwaves. In our preliminary experiment we observed that ratio of reflected signals of 8 GHz between aluminum plate and pellet of carbon beads was 1700/100 mV. This made us interested to study absorption property of microwave by carbon nanomaterials. Thus, as an extension of our previous work[11–14] we report here the study of microwave absorption properties (in the range of 8 GHz to 12 GHz) of carbon nano-materials, synthesized from camphor, by pyrolyzing its vapour at various temperatures in the range of 700 °C to 900 °C.

*Author to whom correspondence should be addressed.

Table I. 9 orthogonal set of experiment designed to find the best condition to get carbon nanomaterials showing best microwave absorption in the range of 8–12 GHz. Values in bracket shows the magnitude of absorption peak for the wavelength obtained with each film.

Variables	L1	L2	L3	L4	L5	L6	L7	L8	L9
Catalyst	Fe	Ni	Co	Ni	Co	Fe	Co	Fe	Ni
Temperature (K)	1023	1123	1273	1023	1123	1273	1023	1123	1273
Absorption wavelength (GHz)	11.5(0.7)	11.5(0.6) 11(0.25)	9.5(0.3)	9.5(0.9) 11.5(0.6)	9.5(0.8) Increases after 11.0 GHz	10.5(0.6) Absorbs from 8.5 to 10.0 and then increases up to 12.0	9.5(0.95) 11.5(0.3)	9.5(0.95) 11.05(0.3)	9.75(0.95) 11.5(0.25)

2. EXPERIMENTAL TECHNIQUE

The parameters, which influence the growth of carbon nanomaterials, are many and they also depend upon the type of CNM one is interested in. Since we were not sure which form of carbon nanomaterials would be most suitable for the microwave absorption, the variable parameters for the preparation of carbon nanomaterials became very large. Hence it was decided to fix some of the variable parameters like, type of precursor (i.e., camphor), type of gas (i.e., nitrogen), its flow rate (i.e., 5 ml/minute) and vaporizing temperature of camphor (523 K). For the first set of experiment only two variables were thus selected: nature of catalyst (nanosize (~50 nm) powder of Fe, Co and Ni), temperature of pyrolysis (1023 K, 1123 K and 1273 K). Considering these parameters a set of 9 experiments were designed (Table I).

Chemical vapor deposition was carried[15] out inside a fused silica reaction tube of 1 m long and inner diameter 0.25 m, housed within two horizontally placed independent cylindrical furnaces (Fig. 1). With this arrangement, the reaction tube was made to have two different temperature zones: zone F1 (vaporizing zone) and zone F2 (CVD zone). Vaporization of camphor was carried out at zone F1 under constant temperature (523 K). A constant supply of camphor vapor was admitted into the CVD zone by a predetermined flow rate of a carrier gas (15 ml/min), controlled through a leak valve, a flow controller and a gas source. Catalysts (Fe, Co, and Ni powder size ~50 nm) and the substrates were placed in a holder within the CVD zone. Substrates (alumina plate 1×2 cm^2) were cleaned prior to the transfer into the CVD zone. Prior to each deposition, the reaction chamber was flushed for 30 minutes with carrier gas to ensure removal of oxygen.

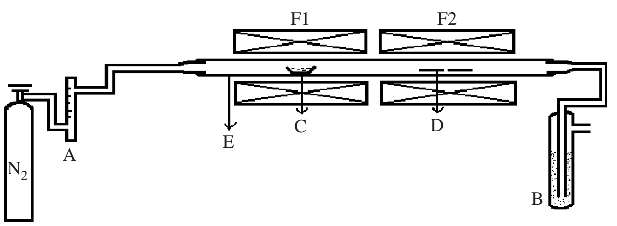

Fig. 1. A self-explanatory thermal CVD set-up A—flow meter, B—water bubbler, C—boat containing camphor, D—substrate, E—quartz tube, F1 and F2—electric furnaces, N_2—N_2 gas cylinder.

About 1 g of camphor was vaporized in the zone F1 (temperature = 523 K) and vapor was admitted into the CVD zone F2 (temperature: 1023–1273 K) where deposition was carried out for 30 minutes. After deposition, tube was slowly cooled down to room temperature. Thin film of carbon nanomaterials prepared under different conditions as mentioned in Table I, were then examined by SEM and TEM. These films were then used to study the absorption of microwave in the range of 8–12 GHz.

3. MICROWAVE MEASUREMENT

Thin films prepared by chemical vapour deposition method had thickness in the range of 3–5 μm. These films were tested for their microwave absorption properties by the help of HP 8510 network analyzer system, which consists of HP 8510A network analyzer, the HP 8340A synthesized sweeper and the HP 8515A S-parameter test set. The system was first calibrated and checked by a standard matched load, then sample was kept inside the slot of X-band Wave-Guide and Return Loss of the deposited sample was measured. The range of frequency used for these measurements were from 8 GHz to 12 GHz.

4. RESULTS AND DISCUSSION

4.1. Microwave Absorption Study

Samples of carbon thin film obtained by depositing carbon over alumina plate under the various conditions as given in Table I were examined for their microwave absorption in the range of 8–12 GHz. Reflection loss or Return loss was calculated using the equation:

$$\text{Reflection loss [dB]} = 20 \log_{10} \frac{E_1}{E_2}$$

where E_1 and E_2 are input and output electric fields of the electromagnetic wave, respectively.

The results of absorptions are shown in Figure 2. It is clear from the graphs that while film obtained from L6 seems to absorb from 8.5 GHz to 10.05 GHz, and then decreases to 11.5 GHz. Films obtained from experiments number L3, L4, L5, L7, L8, and L9 gives maximum absorption at 9.5 GHz only and L3 gives small absorptions at 11.0 GHz. Films obtained from L1, L2, and L4

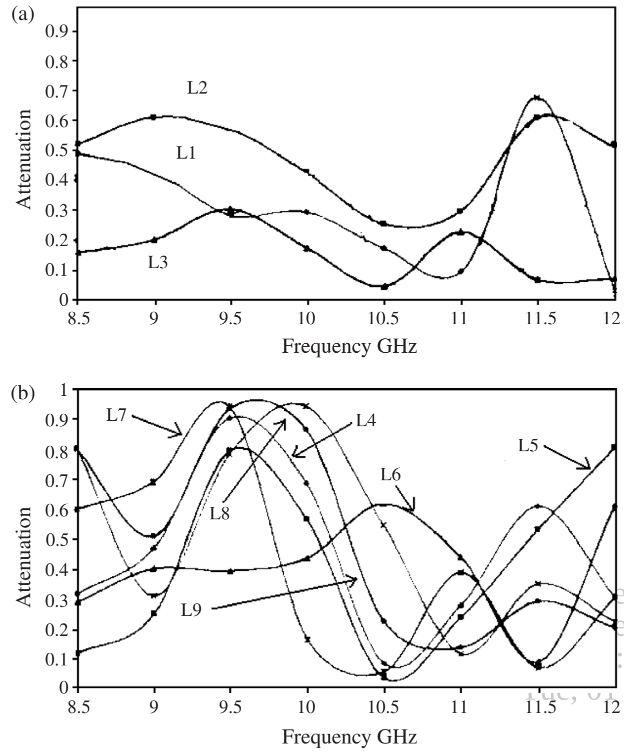

Fig. 2. Absorption of microwave obtained from various samples prepared under the condition given in Table I.

gives maximum absorption at 11.5 GHz. Amongst all these experiments, it seems that experiment nos. L2 and L6 could be developed to get absorption from 8 GHz top 11.5 GHZ. It is important to note that both these films were prepared under iron and nickel catalyst.

Moreover, the examination of morphology of all films prepared under the condition mentioned in Table I suggested that those films with beads like shape showed the tendency of giving higher absorption and in special for condition L6. Hence it was decided to prepare thin film of carbon nanobeads from camphor with use of ferrocene as inner catalyst, as our previous experience has suggested that beads can be formed with ferrocene mixed with camphor.[13] Hence in an another set of experiments, carbon nanobeads were prepared by adopting the experimental technique as discussed in this paper but using pyrolyzing temperatures 600 °C, 750 °C and 900 °C. Films deposited over alumina plate prepared under these conditions were examined for their absorption property of microwave in the range of 8–12 GHz (Fig. 3).

It was observed that microwave absorption increases with the samples deposited at higher temperature. It is clear from these graphs that film produced at 900 °C shows the maximum absorption from 8–12 GHz (in the range of 25–35 dB). As compared to previous absorption (L9, Fig. 2) this also shows (Fig. 3) relatively higher absorption at 8 GHz and 11.5 GHz.

Examination of SEM/TEM photographs of films deposited at these three temperatures suggested that as the temperature of pyrolysis increased interspacing between

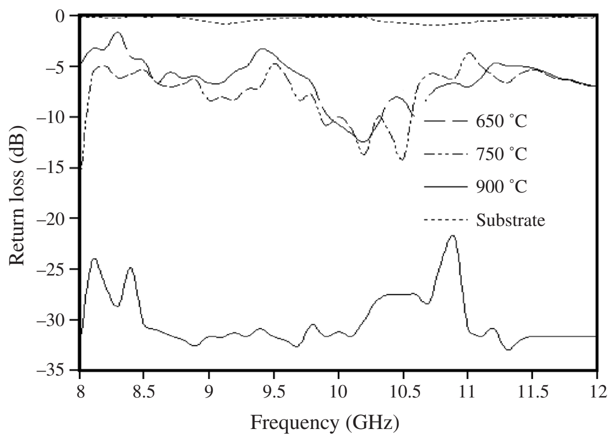

Fig. 3. Plot of return loss versus X-band frequency for three carbon films deposited at different pyrolysis temperature on alumina substrate and a bare alumina substrate. The absorption by alumina substrate is also shown by dotted line to examine the variation in absorption of substrate vis-à-vis carbon thin film.

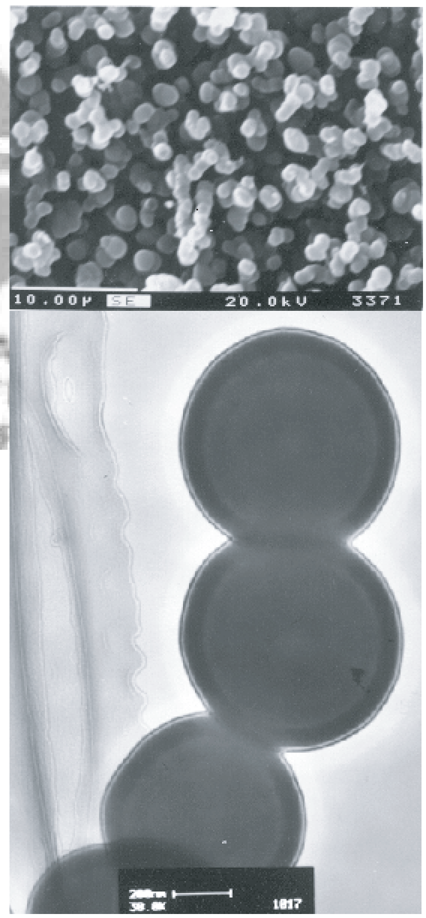

Fig. 4. Top figure is SEM micrographs of carbon nanobeads deposited by pyrolysis of camphor in presence of ferrocene at 900 °C. Bottom micrograph is of powder scratched from the thin film deposited over alumina plate.

RESEARCH ARTICLE

Table II. Resistivities of samples deposited at different pyrolysis temperature.

Pyrolysis temperature (°C)	Resistivity (10^{-3} $\Omega \cdot m$)
600	4.173
750	1.805
900	0.143

granular nanobeads increased to about 2 μm for film deposited at 900 °C (Fig. 4).

4.2. Resistivity Study

In order to understand the role of conductivity of film vis-à-vis absorption of microwave, room temperature resistivities of the samples prepared under three different temperatures were measured (Table II). It is found that resistivity decreases significantly with increase in pyrolysis temperature. Since microwave absorption is better for film prepared at 900 °C suggest that resistivity of the film should also be as low as possible.

5. CONCLUSIONS

These investigations of microwave absorption studies suggest that carbon films can be used as microwave absorber effectively. Moreover, it may also be possible to prepare carbon films, which could absorb a specific wavelength. It is found that these samples are good absorber for 9.5 and 11.0 GHz wavelengths. Carbon nanobeads prepared at 900 °C are good absorber (~25–35 dB) for wavelengths 8–12.0 GHz.

References and Notes

1. S. Ruan, B. Xu, H. Suo, F. Wu, S. Xiang, and M. Zhao, *J. Magn. Magn. Mater.* 212, 175 **(2000)**.
2. H. S. Cho and S. S. Kim, *IEEE Trans. Magn.* 15, 3151 **(1999)**.
3. P. Singh, V. K. Babbar, A. Razdan, S. L. Shrivastava, and T. C. Goel, *Mat. Sci. Eng. B* 78, 70 **(2000)**.
4. C. Y. Lee, H. G. Song, K. S. Jang, E. J. Oh, A. J. Epstein, and J. Joo, *Synthetic Metals* 102, 1346 **(1999)**.
5. S. Courric and V. H. Tran, *Polymer* 39, 2399 **(1998)**.
6. S. Sugimoto, K. Okayarua, S. Kondo, Hiroyasu Ota, M. Kimura, Y. Yoshida, H. Nakamura, David Book, T. Kagaatani, and M. Homma, *Mater. Trans. JIM* 39, 1080 **(1998)**.
7. G. Lu, X. Li, and H. Jiang, *Composites Sci. Technol.* 56, 193 **(1996)**.
8. C. Y. Huang and J. Pai, *Eur. Polym. J.* 34, 261 **(1998)**.
9. N. C. Das, D. Khastgir, T. K. Chaki, and A. Chakraborty, *Composites A* 31, 1069 **(2000)**.
10. X. Luo and D. D. L. Chung, *Composites B* 30, 227 **(1999)**.
11. Kingsuk Mukhopadhyay, Kalaga Murali Krishna, and Maheshwar Sharon, *Phys. Rev. Lett.* 72, 3182 **(1994)**.
12. Kingsuk Mukhopadhyay, Kalaga Murali Krishna, and Maheshwar Sharon, *Carbon* 34, 251 **(1996)**.
13. Maheshwar Sharon, Kingsuk Mukhopadhyay, Kiyoshi Yase, Sumio Iijima, Yoshinori Ando, and Xinluo Zhao, *Carbon* 36, 507 **(1998)**.
14. Maheshwar Sharon, N. Sundarakoteeswaran, P. D. Kichambre, Mukul Kumar, Yoshinori Ando, and Xinluo Zhao, *Diamond Rel. Mater.* 8, 485 **(1999)**.
15. Maheshwar Sharon, Debabrata Pradhan, Yoshinori Ando, and Xinluo Zhao, *Curr. Appl. Phys.* 2, 445 **(2002)**.

Received: 5 February 2005. Accepted: 3 May 2005.