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Microwave Absorption Characteristics of Carbon Nanotubes

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1. Introduction

Radar-absorption materials (RAM) have a growing and widespread applications in broadcasting and television, radar technology and microwave dark-room. In particular, in military applications, stealth weapons exhibit crucial effect in war. The research of RAM has been accelerating with the stealth technology and significant progresses have been achieved. The investigations of RAM began before War II. It has experienced a process from traditional RAM to advanced RAM represented by nano-RAM. These advanced RAM mainly include nano-metals and alloys, nano-oxides, nano-SiC, nano-ferrite, nano-graphite, nano-SiC, nano-SiN, nano conductive polymers and carbon nanotubes (CNTs) [1]. Nano RAM has light-weight, excellent compatibility and broad bandwidth, and considered as important potential RAM for industrial application. CNTs are a very important RAM. They possess high strength and toughness [2,3], excellent electrical and thermal conductivity [4-7]. The investigations of microwave absorption of CNTs had gained a big momentum due to its potential applications in the stealth technology of aircrafts, military equipment and microwave dark-room..

Cao[8] investigated microwave absorption properties of CNTs-polyester and achieved promising results with the absorbing peak value of -14 dB and bandwidth of 10.50 GHz ($R < -5\text{dB}$). Sheng [9] tested the radar wave absorption properties of CNTs coated with nickel. The results show the increase of bandwidth, but the absorption peak decreases. The peak and bandwidth of the RAM of CNTs coated with nickel reach -11.85dB and 2.23 GHz ($R < -10\text{dB}$). Lin [10,11] investigated the microwave-absorption properties of Co-filled and Fe-filled carbon nanotubes respectively. The results indicate that the microwave-absorbing properties of Co-filled CNTs was improved. The maximum reflection loss is about -39.32 dB and the bandwidth corresponding to the reflection loss below -10 dB is 3.47 GHz. With increasing thickness, the maximum reflection loss shifts to lower frequency. The Fe-filled CNTs also show excellent microwave absorption properties. Che[12] investigated the properties of microwave absorption and electromagnetic interference shielding of Fe-filled CNTs. The results shows the microwave absorption and shielding effect was enhanced substantially. The shape of Fe filled into CNTs is largely related to the shielding effect and microwave absorption. Zhao[13] investigate the Microwave absorbing property Ni-coated and Ag nanowires filled carbon nanotubes. CNTs were coated with nickel by an electroless plating technique, CNTs were filled with Ag nanowires via a wet chemical method and epoxy was used as substrate materials.

The microwave absorption properties of the filled CNTs were improved compared with the unmodified CNTs/epoxy composites. Microwave absorption peaks of Ni-coated CNTs/epoxy composites moved to the higher frequencies. For the Ag nanowires filled CNTs, the absorption curve illustrates that reflection loss of the corresponding composites is below -10 dB in the range of 7.2–9.0 GHz, and the minimum value is -19.19 dB at 7.8 GHz. The microwave absorbing peak of the composites moves to the low frequency by filling the Ag nanowires into the CNTs. This shows that the absorption peak frequency of the CNTs/epoxy composites can be manipulated easily by plating different nickel coatings onto the surface of CNTs or filling Ag nanowires into CNTs. It was considered that the microwave absorptions of composites containing CNTs or Ag nanowires filled CNTs result mainly from dielectric loss rather than magnetic loss. In contrast, the microwave absorption of Ni-coated CNTs/epoxy composites was attributed to both dielectric and magnetic losses. Li[14] used multiwalled carbon nanotubes (MWCNTs) and carbonyl iron (CI) as composite microwave absorption agents to investigate the microwave absorption properties of the composite materials. The results show the microwave absorption of the composites was substantially enhanced by adding as little as 1wt% of MWCNTs. The absorption peak of composite reached -22.2 dB. The R (dB) values less than -20 dB was broadened from 1.2 (from 14.2 to 13.0 GHz for pure CI composites) to 2.4 GHz (from 13.6 to 16.0 GHz for the MWCNTs added CI composites) at the whole 2–18 GHz band. The enhancement of the microwave absorption was considered from originating from the combination of MWCNTs and pure CI particles. Zhao[14] employ γ -ray to radiate the composite microwave absorption agent to improve electromagnetic properties of Ni-coated CNTs and achieve a positive results. The prepared MWCNTs/Ni composite was demonstrated to be a ferromagnetic material, and the relative complex permeability and permittivity were studied. The reflection loss of MWCNTs were greatly improved in the frequency range between 3.8 GHz and 18 GHz, with a maximum absorption of -7.2 dB at 6.4 GHz. Zhan[15] employed Typical hydrothermal process to synthesize CNTs/Fe₃O₄ inorganic hybrid material for microwave absorption agent and measured the complex permittivity and permeability of the hybrid materials. The electromagnetic properties were greatly enhanced. Wang[16] prepared a new three element hybrid materials for microwave absorption. The iron-filled carbon nanotubes were coated with FeCo by an electroless plating method to prepare the microwave absorption materials and value the microwave absorption characteristics of them. The results demonstrate that the soft magnetic characteristics of iron-filled carbon nanotubes can be improved after being coated with FeCo alloy nanoparticles which results in more effective microwave absorption. Peng[17] used number simulation method to value the microwave characteristics of MWCNTs. The study focuses on the dielectric properties of CNTs/polymer composite with low CNTs concentration at low-frequency range of 50 MHz–3 GHz. Kim[18] investigated the microwave absorption characteristics of the sandwich constructions composed of CNTs/epoxy and PVC foam. The results show the reflection loss of the hybrid composite structure obtained from the free space measurement system in the X-band frequency range with the absorbing bandwidth of -10 dB was 3.3 GHz (8.2–11.5 GHz) and the maximum and minimum EM absorption rate were 97% and 84%, respectively. Zhang[19] investigated the dielectric, magnetic, and microwave absorbing properties of Sm₂O₃-filled multiwalled carbon nanotubes (MWCNTs). The complex permittivity and permeability were measured at a microwave frequency range of 2–18 GHz. Sm₂O₃ nanoparticles encapsulated in the cavities of MWCNTs enhance the magnetic loss of MWCNTs. The calculated results indicate

that the bandwidth of the modified MWCNTs is much broader than that of unfilled MWCNTs. The maximum reflectivity (R) is about -12.22 dB at 13.40 GHz and corresponding bandwidth below -5 dB is more than 5.11 GHz. With the increase of thickness, the peak of R shifts to lower frequency. The multiple absorbing peaks appeared in the Sm_2O_3 -filled CNT composites, which helps to broaden microwave absorbing bandwidth. Rosa[20] investigated the multiphase composite materials filled with multiwall carbon nanotubes (MWCNTs), short nickel-coated carbon fibers and millimeter-long carbon fibers with various weight fractions. The effective complex permittivity of several composite samples is measured in the frequency range from 8 GHz to 18 GHz. The obtained results show that the addition of the MWCNTs into the mixture allows tuning the EM properties of the composite filled with the short nickel-coated fibers. Numerical simulations are also performed. The best performing screens in the Ku-band have thicknesses of about 2.13 mm and 1.57 mm, minimum reflection of about -73 dB and -45 dB and bandwidth of 6 GHz and 5 GHz, respectively. Fan[21] prepared CNT/polymer composites and measured the electromagnetic characteristics and microwave absorption properties of them in a frequency band of 20-18GHz. The test results demonstrated that the maximum absorbing value reached 17.61 dB and 24.27 dB with a loading of 4wt% and 8wt% of CNTs and the corresponding absorbing peak at 7.6 GHz, 15.3 GHz respectively. The dielectric loss was considered as main attribution to the microwave absorption of CNTs composites rather than magnetic loss.

Although many progresses have been achieved, the bandwidth and peak of RAM need to be improved further and enhanced for military and civil applications. In this paper, the microwave absorption characteristics of CNTs were further investigated. The raw MWCNTs, doped MWCNTs and aligned MMCNTs were used as microwave absorption agents and epoxy resin was used as matrix to prepare the composites to measure the properties of microwave absorption of CNTs.

2. Experiment

2.1 Synthesis of carbon nanotubes

2.1.1 Growth of carbon nanotubes

Carbon nanotubes were grown by the catalytic decomposition of hydrocarbon precursor gas such as acetylene, ethylene, ethanol or methane. In this investigation, liquified petroleum gas (LPG) was used as carbon sources. Metal catalysts mainly include nickel, cobalt, iron, or a combination of them. The metal catalyst nanoparticles can be fabricated by sol-gel, co-deposition processes. The size of catalyst particles has big impact on the diameters of the nanotubes that are to be grown. The smaller particle grows smaller diameter of carbon nanotubes. In the paper, iron was employed as catalyst which plays a crucial role in the nucleation and growth of carbon nanotubes in the thermal chemical vapor deposition (CVD) process. Nanotubes grow at the sites of the metal catalyst. The carbon-containing gas is broken apart at the surface of the catalyst particle, and the carbon is transported to the edges of the particle, where it forms the nanotubes. The substrates of catalyst include diatomite, MgO or Al_2O_3 to increase the surface area for higher yield of the catalytic reaction of the carbon feedstock with the metal particles. The removal of the catalyst support commonly employed an acid treatment method, which sometimes could destroy the original structure of the carbon nanotubes. In this investigation, carbon nanotubes themselves were used as substrates which result in higher purity of as-produced carbon nanotubes. The details of the MWCNTs preparation method [22] were described elsewhere.

2.1.2 Growth of aligned carbon nanotubes

Ferrocene was dissolved in solution of xylene used also as the carbon source. The flat quartz glass substrates were put on the middle of the electrical furnace. The furnace was heated to about 850 °C. Argon gas was introduced to the furnace to eliminate air and then a mixture gas of argon and hydrogen was introduced as the carrier gas. The solution of ferrocene and xylene was injected into furnace and was vaporized. The vapor then went into the reactive area where the ACNTs grew on surface of quartz glasses.

2.1.3 Preparation of the composites

2.1.3.1 Preparation of MWCNT/epoxy composites

Carbon nanotubes with different loading weight of CNTs were respectively added into epoxy resin and are were sufficiently mixed by high-speed stirring dispersion and ultrasonication. The mixture was smeared onto an aluminum plate layer by layer until the thickness of the composites reached 4mm.

2.1.3.2 Preparation of ACNTs/epoxy composites

The films of ACNTs were peeled off from the quartz glasses. Then the films were put on the surface of a aluminum plate with size of 180X180mm.and were fixed by epoxy resin to prepare the samples. After cured, the microwave absorption characteristics of the composites were measured.

2.2 CNTs characterization

In this work, a FEI Quanta 200 scanning electronic microscopy (SEM) and a Hitachi H-600 transmission electronic microscopy (TEM) were used to observe the structure and morphology of CNTs. A TG/DTA Pyris diamond was employed for thermogravimetric analysis to ascertain the purity of carbon nanotubes.

2.3 Microwave absorption characteristics of CNTs

Microwave absorption characteristics of CNT/epoxy composites are measured through radar absorption materials (RAM) measuring system of arch method reflectivity.

3. Results and discussion

Fig.1 is a TEM image of CNTs and Fig.2 is a SEM image of CNTs. The images demonstrate the CNTs are multiwalled carbon nanotubes(MWCNTs) and have a diameter of 10-30nm with a length of about several micrometers. The images also show that the MWCNTs were entangled with each other to become clusters that are the the most main challenge to meet for effective industrial applications for most fields. Specially for composite materials. Fig.3 shows the TEM image of dispersed ACNTs. Fig.4 and Fig.5 show SEM images of Low-magnifying and high- magnifying SEM image of ACNTs. and Fig.6 is micro-image of ACNTs shot by common camera. Fig.7 demonstrates the top morphology of ACNTs.

Fig.4 and Fig.5 show that ACNTs were vertically grown on the substrates. The growth orientation are contained each other in growth and they only can vertically grown on the surface of substrates. So the carbon nanotube arrays were shaped. Fig.6 shows the top of ACNT film looks like the brushwood. Fig.3 shows that CNTs in ACNTs are multi-walled carbon nanotubes (MWCNTs). The MWCNTs have a diameter of 30~50 nm.

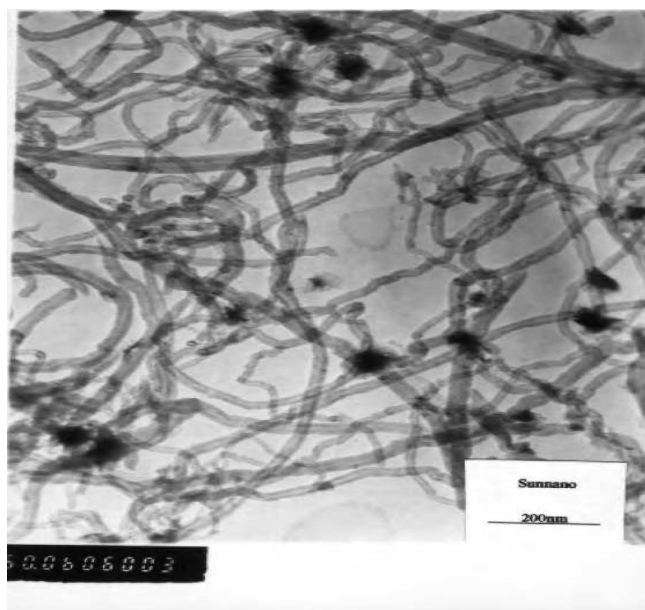


Fig. 1. TEM image of MCWNTs

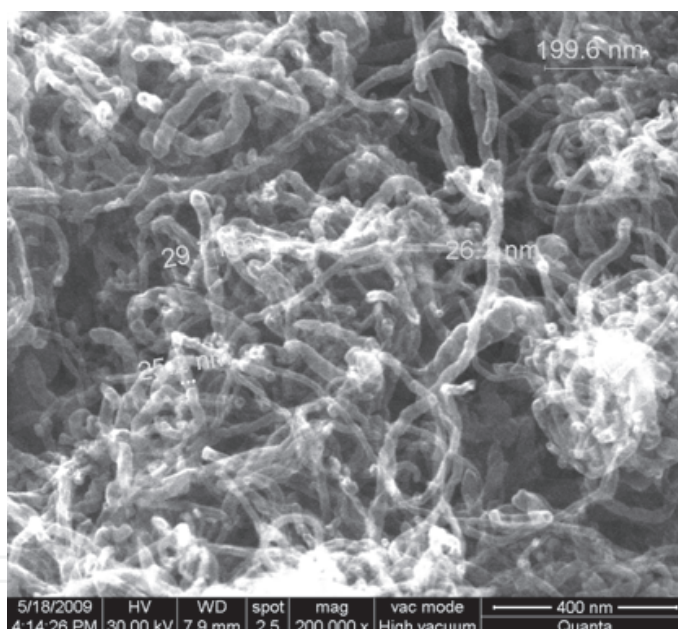


Fig. 2. SEM image of MCWNTs

The purity of CNTs reaches over 95%. The thermogravimetric analysis (TGA) of MWCNTs was shown in Fig.8. The MWCNTs have high thermal stability. It can be found that weight was lost slowly from 50 to 550°C, corresponding to the loss of little water and a few amorphous carbon. At the temperature range from 550 °C to 700, the weight decreased sharply to 2.20 wt%, indicating that the combustion of the MWCNTs started at 550°C. Also a thing to note is that the curve slope maintained almost the same value from 550 to 700°C. It is illustrated that the MWCNTs were combusted at a constant speed, suggesting that the MWCNTs reached to a high purity at 550°C. After 700°C, the weight of the sample remains unchanged. The remainders may be assigned to catalyst including 2.20 % iron and aluminium metal oxides. Therefore, 550°C can be considered as an optimum temperature to

burn out amorphous carbon particles for CNT purification. The figure demonstrate the purity of measured CNTs reached 97.80%.



Fig. 3. TEM image of dispersed ACNTs

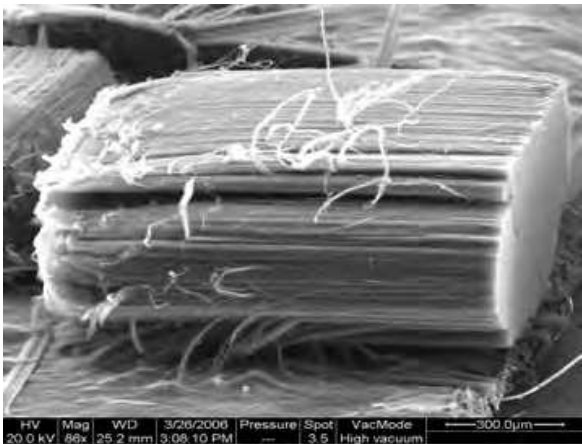


Fig. 4. Low-magnifying SEM image of ACNTs

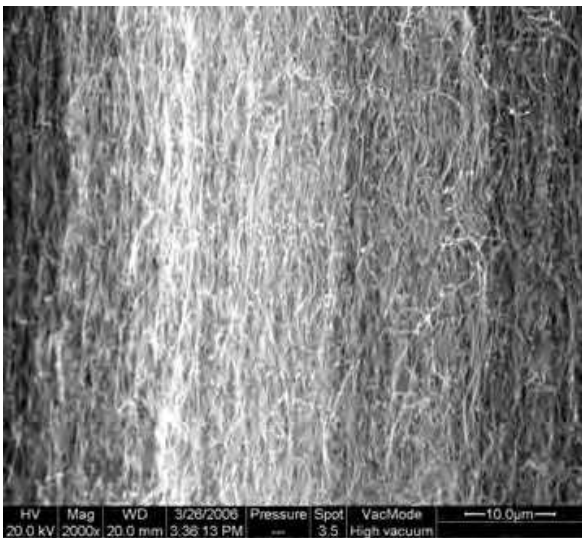


Fig. 5. SEM image of ACNTs

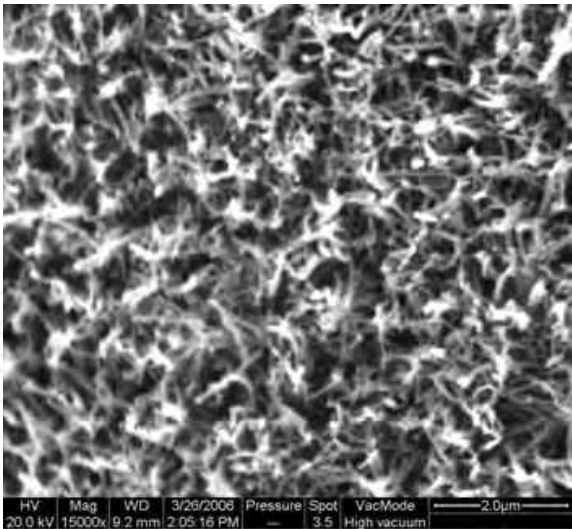


Fig. 6. Top SEM image of ACNTs

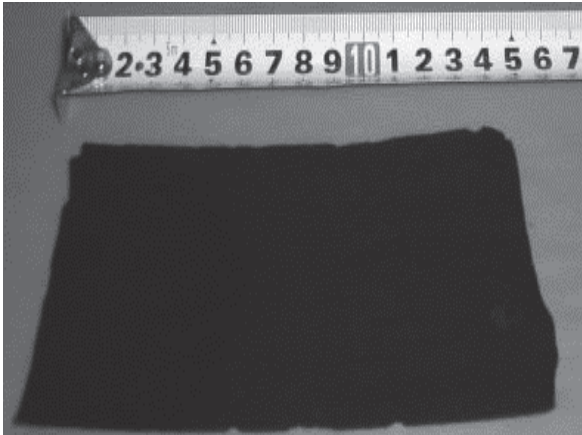


Fig. 7. Macro image of ACNTs

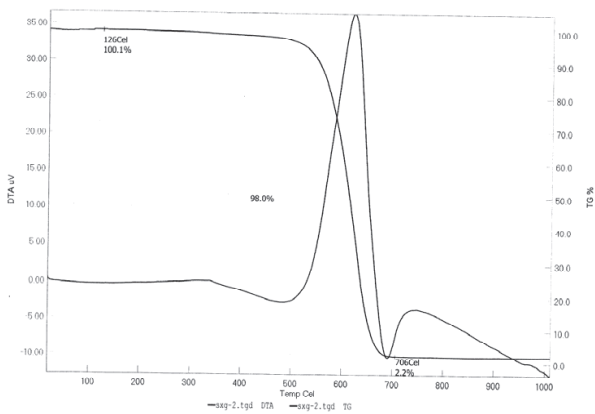


Fig. 8. Thermogravimetric curves of MWCNTs

The Fig.9 shows SEM image of cross-section of CNT/epoxy composites. The figures demonstrate the MWCNTs were well dispersed in epoxy resin matrix but some entangled MWCNTs can still be observed in the epoxy resin matrix.

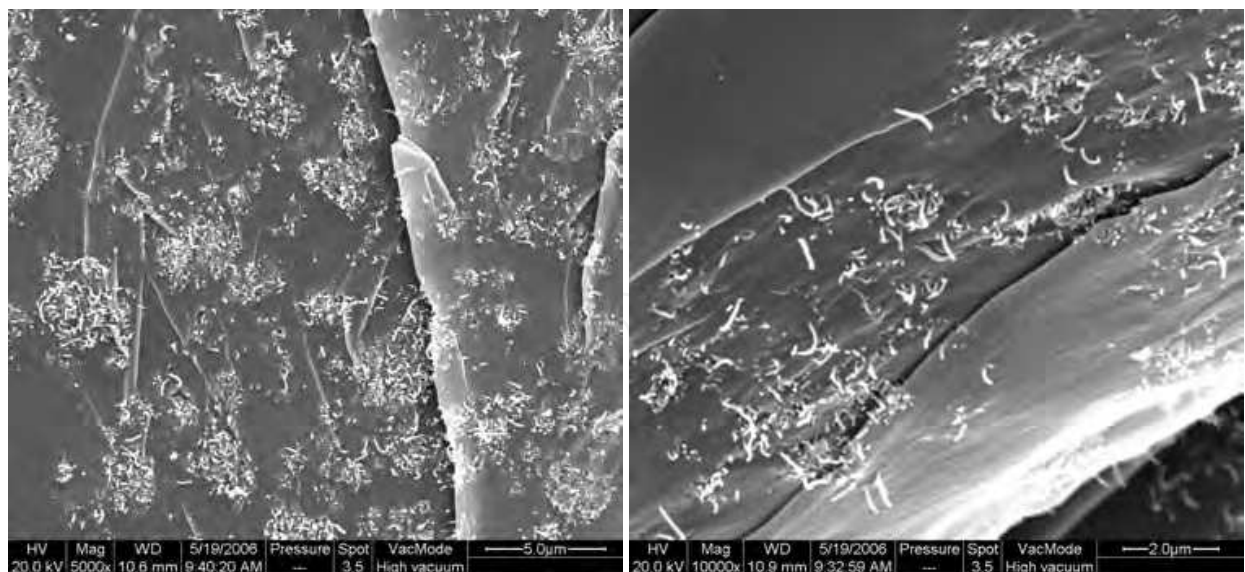


Fig. 9. SEM image of cross-fracture of MWCNT/epoxy composites

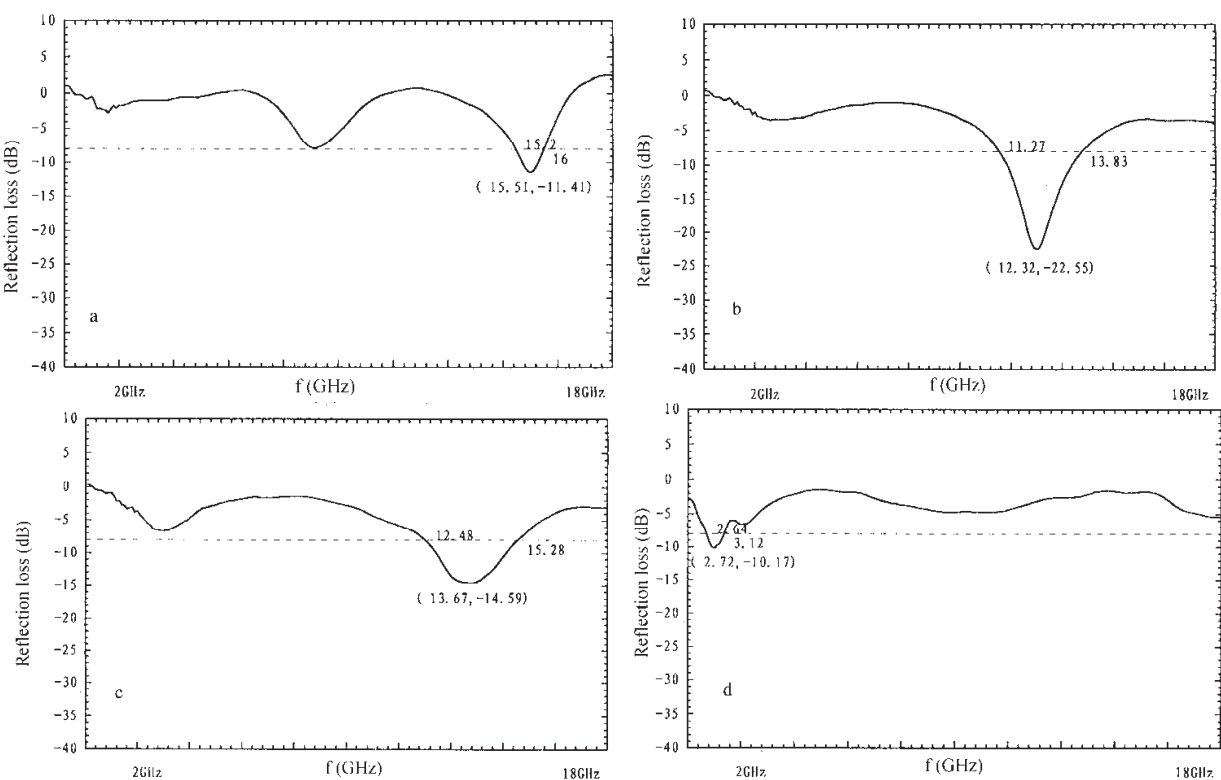


Fig. 10. Microwave absorbing properties of epoxy matrix composites containing CNTs with different loading of (a)5wt%, (b) 8wt%, (c) 10wt% and (d) 20wt%.

Fig.10 shows the reflection loss curves of different MWCNT/epoxy composites. The figures of reflecton loss show carbon nanotubes can effectively absorb radar waves in frenquency band of 2-18GHz.The absorbing peak, absorbing peak value and bandwidth change with different loading of carbon nanotubes. The wave absorption paprameters were showed in table.1. The sample b obtained the largest absorbing peak value of -22.55 dB at 12.32 GHz with a bandwidth of 2.56 GHz ($R<-10$ dB) and 4.16 GHz ($R<-5$ dB) respectively.

The sample c obtained the largest bandwidth of of 2.80 GHz ($R<-10\text{ dB}$) and 6.24 GHz ($R<-5\text{ dB}$) respectively. Another important characteristic, as demonstrated in Fig10a,c,d is double absorbing peaks, The second largest peak value reach -7.73 dB at 9.2 GHz , -6.62 at 4.40 GHz and -5.39 dB at 17.80 GHz for sample a, c,d. The duble absorbing peaks is a peculiar properties of carbon nanotubes which can improve wave absorbing properties and enhance the bandwidth.

| Samples No. | CNT (%) | Depth (mm) | peak value (R/dB) | absorbing peak (f/GHz) | bandwidth (f/GHz) | |
|-------------|---------|------------|-------------------|------------------------|-------------------|--------|
| | | | | | R<-10dB | R<-5dB |
| a | 5 | 8 | -11.41 | 15.51 | 0.79 | 3.2 |
| b | 8 | 8 | -22.55 | 12.32 | 2.56 | 4.16 |
| c | 10 | 8 | -14.59 | 13.67 | 2.8 | 6.24 |
| d | 20 | 8 | -10.17 | 2.72 | 0.47 | 2.56 |

Table 1. Radar waves absorbing properties of CNT/EP composites

Fig.11 shows microwave absorption characteristics of rare earth doped MWCNT composites (a) 5wt% CNT and 1% CeO₂,and (b) 8wt% CNT and 1% CeO₂. The Fig.11 indicated the radar waves absorbing properties of carbon nanotubes were substantially improved after modification by rare earth oxides. The highest absorbing peak of the sample reaches -29.10 dB at 10.88 GHz and the bandwidth reaches 7.68 GHz ($R<-10\text{dB}$) loaded with 1wt% rare earth and 8% carbon nanotubes compared with undoped MWCNT with the absorbing peak value of 12.32dB and bandwidth of 2.80 GHz ($R<-10\text{ dB}$) . The peak value increased by 16.87 dB and bandwidth increased by 4.88GHz .

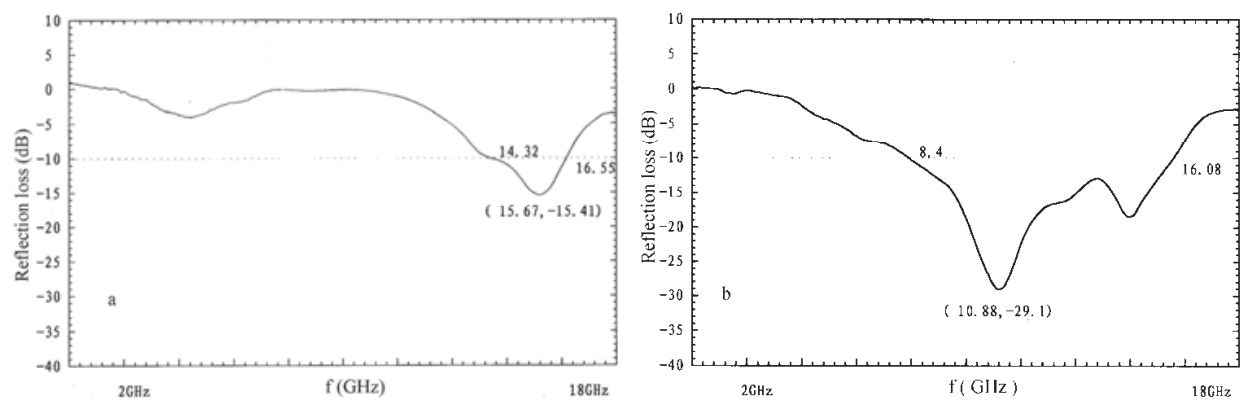


Fig. 11. Microwave absorption characteristics of rare earth doped MWCNT composites.

Rare earth elements are of great importance in magnetic, electronic, and optical materials because of the number of unpaired electrons in their 4f shells. The unique electronic, optical and chemical properties make them useful in radar wave absorption. The rare earth 4f shells were not fully filled. This results in the magnetic moment. The magnetic moment of rare

earth atoms or ions are related with not only the factor g_1 but although angular momentum J . These unique characteristics enhance largely the microwave absorption of CNTs. Fig.12 demonstrates microwave absorption characteristics of aligned carbon nanotubes with different film thickness of (a) 20 μ m, (b) 2mm and (c) 1mm. The microwave absorption parameters were listed in table2.

| Samples | Depth (mm) | Absorbing peak (R/dB) | Absorbing peak (f / GHz) | Frequency | Width |
|---------|---------------|--------------------------|-----------------------------|----------------------|--------|
| | | | | (f / GHz) R<-10dB | R<-5dB |
| a | 0.2 | -15.87 | 17.83 | 4.25 | 6.40 |
| b | 1 | -10.02 | 17.83 | 0.16 | 4 |
| c | 2 | -7.54 | 13.80 | 0 | 6.40 |

Table 2. Microwave absorption parameters of ACNTs

As showed in Fig.4 and Fig.5, the aligned carbon nanotubes were vertically grown on the substrates in orientation. they are restricted in growth orientation and only can vertically be grown on the surface of substrates and so the array of carbon nanotubes was shaped. The carbon nanotubes in array parallels each other and resulted in anisotropy of aligned carbon nanotubes. Fig.6 shows the top of ACNT films looking like the hassock. This morphology is similar to jungle structure of radar wave absorption disguise and is beneficial to microwave absorption[23]. When electromagnetic wave reaches the top surface of ACNTs, the part was reflected back into air and other enters into ACNT jungle. The electromagnetic wave was hold in the hassock of ACNTs and was absorbed and exhausted in repeat reflection.

The microwave absorption curves and absorption parameters of ACNTs show ANTs have excellent microwave absorption properties in higher band in 2~18GHz. The absorbing peak and bandwidth change with different thickness of films of ACNTs. The absorption peak of the sample A reaches -15.87dB at 17.83 GHz and has a bandwidth of 4.25 GHz (R<-10dB) and 6.40GHz (R<-5 dB) . The absorption peak of the sample B reaches -10.02 dB at 17.83 GHz and has a bandwidth of 0.16 GHz (R<-10dB) and 4.00GHz (R<-5 dB) . The absorption peak of the sample C reaches -7.54 dB at13.80 GHz and has a bandwidth of 5.40 GHz (R<-5 dB) .

The test results indicate the absorption peak of ACNTs decreases with the increase of thickness of film of ACNTs. The peak decreases from -15.87 dB (sample A) to -7.54 dB (Fig.c) . The peak is situated at higher band of 2~18 GHz. This may be resulted from the increase reflection rate of microwave on thicker film of ACNTs. The microwave absorption bandwidth of ACNTs also changes with thickness of films of ANTs. The bandwidth of reflection loss less than -10dB decreases from 4.24 GHz (sample A) to zero (Fig.c). The bandwidth of reflection loss less than -5dB first decreases from6.40 GHz (Fig.a) to 4.00 (Fig.b) and then again increase 6.40 GHz (Fig.c). These do not exhibits obvious trend.

Nanomaterials have a new wave absorption mechanism from the effects of small size, surface and quanta size. The quanta size effect brings out the breakage of periodic boundaries and make the characteristics of voice, light, electron, magnet and energetics

changed, so they substantially enhance the properties of microwave absorption of nanomaterials. CNTs have vast surface, high rate atoms of surface and substantive hanging-bonds. These effect of interface polarization and multiple dispersion improve the electromagnetic wave absorption of CNTs. The quanta size effect makes energy level of electrons to be splitted. The gaps of energy just contain microwave energy level ($10^{-2} \sim 10^{-5}$ eV), and so produces a new way of wave absorption. Further more, CNT has two kinds of structures including the symmetry and chirality. The chiral structures enhance the radar wave absorption performance of CNTs. CNTs were dispersed into polymer matrix to form electrically conductive network in polymer. The energy of electromagnetic waves was also attenuated in network resistors, which is similar to the resistive type of wave absorption materials.

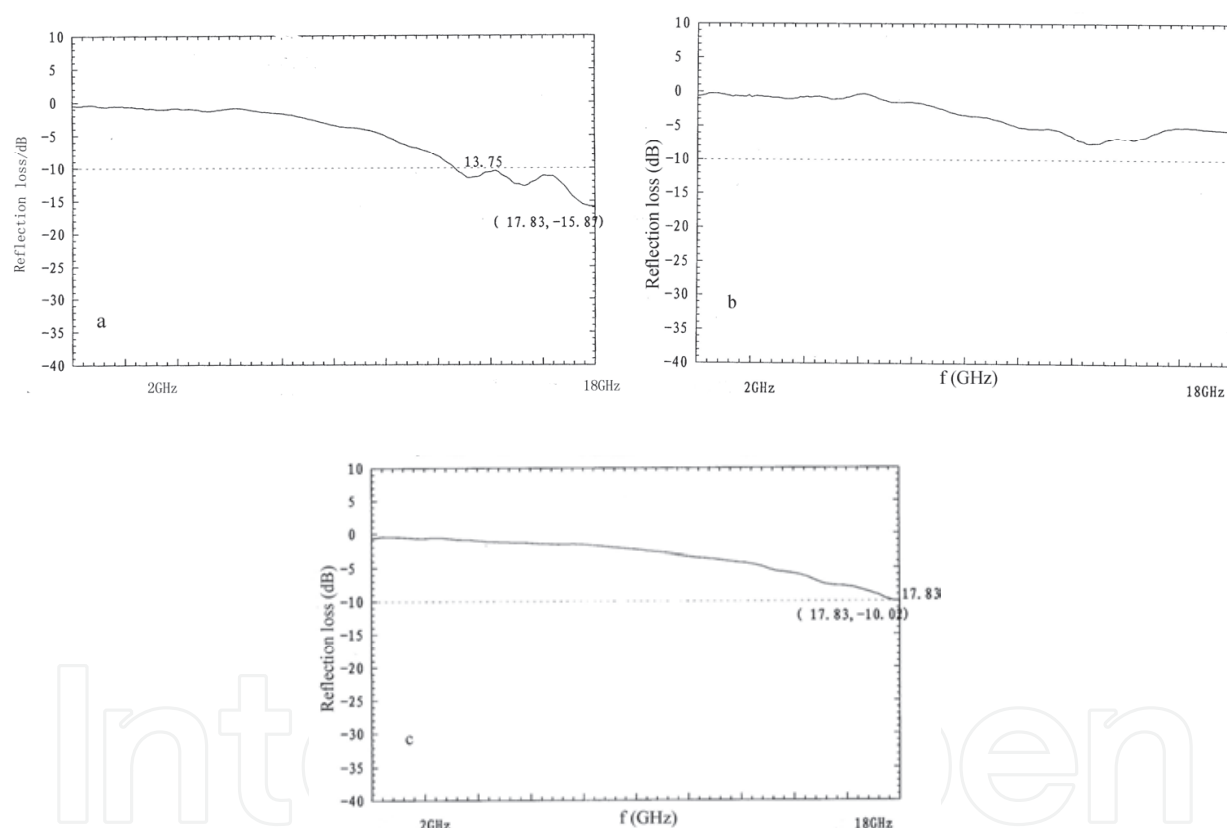


Fig. 12. Microwave absorption characteristics of aligned carbon nanotubes with different film thickness of (a) 20 μm , (b) 2 mm and (c) 1 mm.

Rare earth elements are of great importance in magnetic, electronic, and optical materials because of the number of unpaired electrons in their 4f shells. The unique electronic, optical and chemical properties make them useful in radar wave absorption. The rare earth 4f shells were not fully filled. This results in the magnetic moment. The magnetic moment of rare earth atoms or ions are related with not only the factor g_1 but although angular momentum J . These unique characteristics enhance largely the microwave absorption performance of CNTs.

4. Conclusion

Summary, carbon nanotubes including multiwalled carbon nanotubes, rare earth doped carbon nanotubes and aligned carbon nanotubes were investigated for stealth applications. The reaserch demostrated that carbon nanotubes have good microwave absorption properties. The rare earth doped carbon nanotubes showed better microwave absorption properties. CNTs doped with rare earth demonstrate good improvement of microwave absorption properties. The absorbing peak of the CNTs doped with 1wt% rare earth reached -29.10 dB at 10.88 GHz and the bandwidth reached 7.68 GHz ($R < -10\text{dB}$) loading with 8% carbon nanotubes compared with undopped MWCNT with the absorbing peak value of 12.32dB and bandwidth of 2.80 GHz ($R < -10\text{ dB}$). The peak value increased by 16.87 dB and bandwidth increased by 4.88GHz.

Alinged carbon nanotubes showed good microwave absorption prperties in higher band of 2~18 GHz. The microwave absorption peak value of ACNTs decreased with the increase of thickness of film of ACNTs. The peak value decreased from -15.87 dB to -7.54 dB. The peak is situated at higher band of 2~18 GHz. The microwave absorption bandwidth of ACNTs also changes with the thickness of films of ANTs. The bandwidth of reflection loss less than -10dB reached 4.24 GHz.

Carbon nanotbes demonstrate big potential in industrial applications for microwave absorption. The futher investigation was needed to devolop the applications, specailly for stealth arms.

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Carbon nanotubes are one of the most intriguing new materials with extraordinary properties being discovered in the last decade. The unique structure of carbon nanotubes provides nanotubes with extraordinary mechanical and electrical properties. The outstanding properties that these materials possess have opened new interesting researches areas in nanoscience and nanotechnology. Although nanotubes are very promising in a wide variety of fields, application of individual nanotubes for large scale production has been limited. The main roadblocks, which hinder its use, are limited understanding of its synthesis and electrical properties which lead to difficulty in structure control, existence of impurities, and poor processability. This book makes an attempt to provide indepth study and analysis of various synthesis methods, processing techniques and characterization of carbon nanotubes that will lead to the increased applications of carbon nanotubes.

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