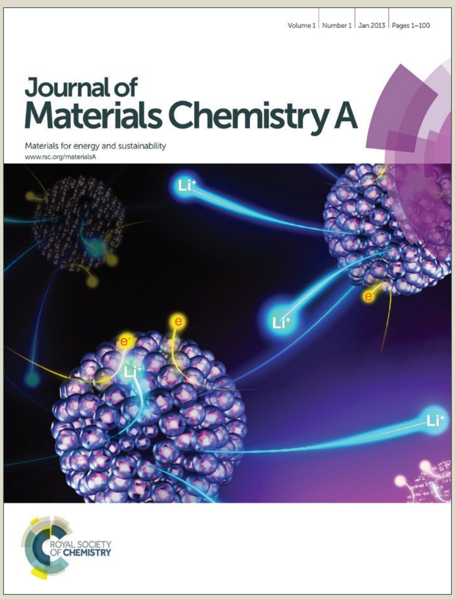

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Microwave shielding properties of Co/Ni decorated single walled carbon nanotubes

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Abstract

Cobalt/nickel nanoparticles decorated single-walled carbon nanotubes (Co/Ni @SWCNTs) were prepared by dc-arc discharge technique. Co/Ni @SWCNTs was characterized by scanning electron microscope, high resolution transmission electron microscope (HRTEM), Raman spectroscope and energy dispersive x-ray analysis techniques. HRTEM results confirmed attachments of magnetic nanoparticles onto SWCNTs with 1.2 nm diameter. A microwave shielding effectiveness value of 24 dB (blocking > 99% radiation) by 1.5 mm thick sample in the frequency range of 12.4-18 GHz was observed. In order to understand the mechanism of shielding; dielectric and magnetic attributes of the shielding effectiveness of Co/Ni @SWCNTs have been evaluated. Eddy currents and natural resonances due to the presence of magnetic nanoparticles, electronic polarization and their relaxation, interfacial polarization and unique composition of the shield contributed significantly in achieving good shielding effectiveness. The observed microwave shielding crossed the limit (20 to 30 dB) required for commercial applications which suggests that these nanocomposites are promising microwave shielding material in the Ku band.

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Introduction

Carbon based composites have gained significant popularity as microwave shielding materials because of their low density, facile synthesis and ease of processing. Recently, research on carbon containing nanomaterials has taken a leap [1]. In general, there are two practical ways for electromagnetic wave shielding by materials: the first is to protect certain components from the radiation by reflecting the waves; another is to reduce the refection and increase the absorption by incorporating dielectric or magnetic particles into it[2]. On account of their ability to reduce electromagnetic wave pollution and radar signatures, microwave shielding materials which can absorb in the frequency range of 1-20 GHz are in high demand for civil as well as military applications[3].

For microwave absorption in high frequency range (over gigahertz) soft magnetic metallic materials are considered better than traditional ferrite absorbers. Nevertheless, the relative complex permeability of magnetic metallic materials decreases due to eddy current induced by electromagnetic waves. Thus, it may be better to use the isolated metallic particles having sizes smaller than the skin depth of electromagnetic waves into the materials[4]. Therefore, research on various materials; e.g., Fe_3B/Y_2O_3[5], rare earth lanthanum nitrate doped amorphous carbon nanotubes (CNTs)[6], Fe/CNTs [7, 8] and $CoFe_2O_4$/CNTs[9] composites have been carried out by various researchers.

Recently, MWCNTs [10-14] have been explored as a potential candidate for developing microwave shielding materials and thus have open a new avenue for investigating applications of other carbon materials in shielding. The smaller diameter, higher aspect ratio, good conductivity and higher mechanical strength of CNT make them an excellent candidate for producing conductive composites useful for high performance EMI shielding materials at low filling [11, 12]. The mechanical properties of CNTs have drawn intense interest for their usefulness as reinforcement material in composites.

Shielding effectiveness (SE) of any shield is governed by its dielectric attributes, magnetic permeability, thickness and frequency of incident radiation. The absorption loss depends on the value of ε_r/μ_r, and the absorption loss is maximum when, $\mu_r = \varepsilon_r$ [15]. The presence of magnetic materials helps in matching of ε_r and μ_r which is necessary for enhancing the absorption of EM wave. However, to the best of our knowledge, SWCNT in EMI SE applications has not been thoroughly explored till date.

Single walled carbon nanotubes (SWCNTs) have been explored for several applications; energy storage and harvesting, electronics appliances, and biomedical applications. But, their exploration in microwave absorption and microwave shielding is still limited. Microwave absorption properties of SWCNTs/polyurethane composites in the frequency range of 2-18 GHz have been studied by Liu et al [16]and a maximum absorption value of 22 dB for 5 wt. % SWCNTs in polyurethane has been observed. Huang et al. have reported EMI-SE of 18 dB for composites with 15 wt% small SWCNTs and 23–28 dB for composite with 15 wt% long SWCNTs in the frequency band of 8–12.4 GHz [17]. Li et al[18] reported EMI shielding properties of SWCNT epoxy composites and showed EMI shielding effectiveness of 15-20 dB in the 500 MHz to 1.5 GHz and 49 dB at 10 MHz frequency. Wadhwan et al [19] have reported better microwave absorption properties of unpurified SWCNTs possessing magnetic Fe nanoparticle impurities (due to the cooperative effect). Srivastava et al. reported flexible, non corrosive and light weight nickel nanoparticle multiwalled carbon nanotubes (MWCNT)–polystyrene composite films as good microwave absorbing material in the frequency range of S band (2-4 GHz) [20]. Recently, Co decorated MWCNTs has also been used as a microwave absorbing materials[21]. To the best of our knowledge, there is no study on the microwave absorption or microwave shielding of Co/Ni nanoparticles decorated SWCNTs.

Binary mixtures of active catalysts such as Ni, Fe and Co are frequently used and often observed to display a higher activity than individual elements. Apart from the above, they also prevent or control the coarsening of the catalyst resulting in better quality and higher yield of SWCNTs. Therefore, in this study, Co/Ni decorated SWCNT (Co/Ni @SWCNTs) were synthesized by dc arc discharge technique using mixture of Co and Ni as catalyst. This was done by arcing graphite in the presence of Co/ Ni and their microwave shielding properties have been investigated. It has been shown that even 1.5 mm thick sheet of as-produced Co/Ni @SWCNTs can act as an efficient microwave shielding material.

Experimental

Co/Ni @SWCNTs was synthesized by dc arc-discharge process. A high density graphite block (purity 99.9%) was machined to 10 mm (dia.) solid cylinder and used as cathode. Anode was a hollow graphite cylinder which was filled with Co/Ni and graphite powders. The anode used for arc-discharge was having a length of 100 mm, outer diameter of 8 mm, inner diameter of 6.5 mm, and drilled depth of 80 mm. A mixture of Ni and Co powders (2 at% each , purity >99.9%, average particle size <3 μm, Aldrich) which were used as catalyst were ball milled along with graphite powder and filled into the hole (dia. 6.5 mm) drilled in graphite anode. For maintaining a stable arc-discharge, a uniform gap of 1-2 mm was maintained between the electrodes with the help of a stepper motor. A dc voltage of 20–25 V, current of 100–120 A and helium pressure of 600 torr was used. A schematic for the synthesis of Co/Ni @SWCNTs is shown in Figure 1.

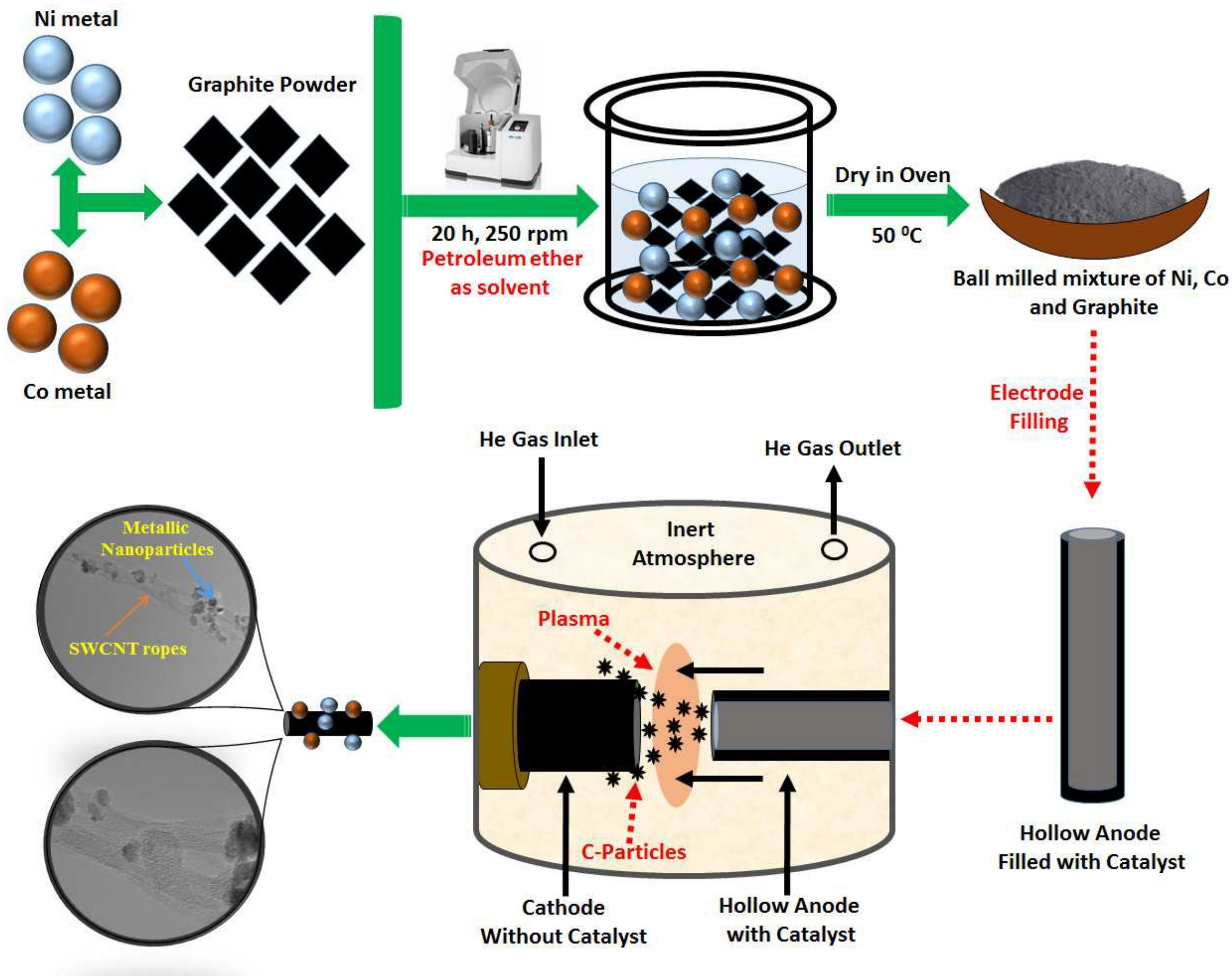

Figure 1. Schematic showing production of Co/ Ni@ SWCNT by dc arc-discharge method

Results and Discussion

Scanning electron microscopic (SEM) image of the grown material shown in Figure 2a reveals the presence of carbon nanotubes. High resolution transmission electron microscopic (HRTEM) images as shown in Figure 2 b and c reveal the presence of metallic nanoparticles over SWCNTs. Raman spectrum of this material indicated a peak at 185 cm^{-1} corresponding to radial breathing mode of SWCNTs (shown in Figure 2d).

According to Araujo et al[22] diameter of SWCNTs can be estimated using the following relation,

$$\omega_{rbm} = 227/d_t \qquad (1)$$

where, ω_{rbm} is the Raman shift (cm^{-1}) and d_t is the tube diameter of CNT (in nm). Figure 2e shows the EDX spectra of @ Co, Ni SWCNTs, where the presence of Co, Ni, carbon and oxygen can be seen. HRTEM image of SWNCTs and SAED pattern of Co/Ni decorated SWCNTs are shown in Figure S1 and S2 (See supporting information).

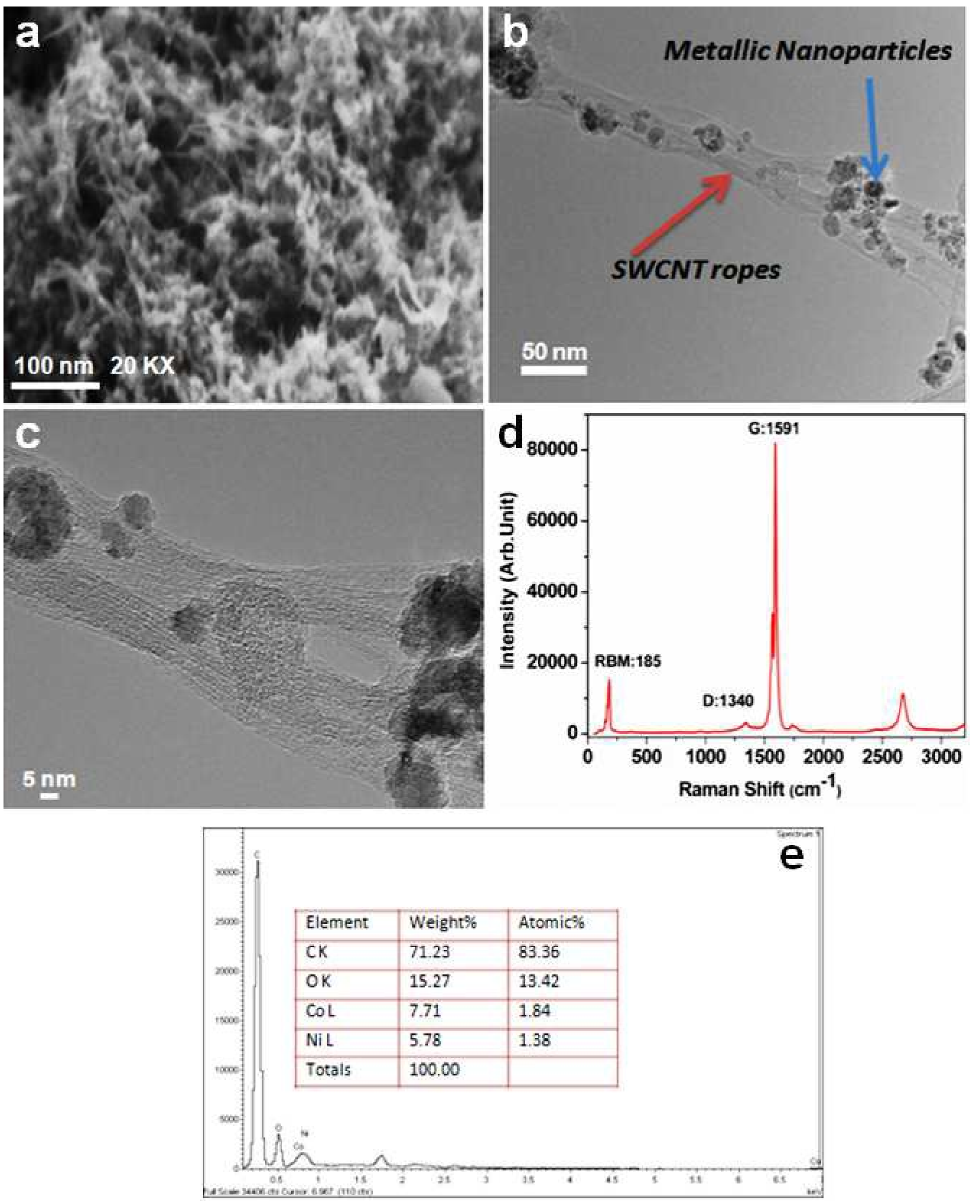

Figure 2. (a) SEM image, (b and c) HRTEM images, (d) Raman spectrum and (e) EDX of Co/Ni@SWCNTs

The room temperature field dependence magnetic properties of Co/Ni @SWCNTs have been studied by plotting the M-H curve and the results are shown in Figure 3a. The saturation magnetization (Ms) value for the Co/Ni @SWCNTs synthesized by arc-discharge method was ~12.25 emug^{-1} at an external applied field of 5 kOe. The M-H curve shows very small value for coercivity and negligible value for retentivity (without any hysteresis loop), indicating a superparamagnetic nature (as shown in inset of Figure 3a). The pristine SWCNT is non-magnetic in nature and therefore the observed magnetization in Co/Ni @SWCNTs is attributed to the presence of high wt% of Co and Ni onto SWCNTs. Furthermore, the observed magnetic behavior of Co/Ni @SWCNTs is supposed to be helpful in achieving higher shielding effectiveness (SE) value suitable for EMI shielding applications.

To further understand the mechanism of EMI shielding, dielectric measurements of the composite have been carried out using Agilent E8362B Vector Network Analyzer. The powder of Co/Ni @SWCNTs was pressed into rectangular shape of 22.8 X 10 mm^2. The rectangular shaped samples with different thicknesses were inserted into the copper sample holder placed between the wave-guides. The S parameters, Sij {S_{11} (S_{22}), S_{12} (S_{21})}[18] were recorded using vector network analyzer (VNA E8263B Agilent Technologies) in the frequency range of 12.4 – 18 GHz (Ku band) via two port measurement techniques. The power coefficients, transmission coefficient (T) and reflection coefficient (R) were calculated using the equations,

$$T = \left|\frac{E_T}{E_I}\right|^2 = |S_{21}|^2 = |S_{12}|^2 \quad (2)$$

$$R = \left|\frac{E_R}{E_I}\right|^2 = |S_{11}|^2 = |S_{22}|^2 \quad (3)$$

and the absorption coefficient can be expressed in terms of R and T as, $(A) = 1 - R - T$.

Absorption efficiency (AE) can be obtained using the relation, AE=A/(1-R)×100%.

EMI SE of any material is the sum of the contributions from the absorption (SE_A), reflection (SE_R) and multiple reflections (SE_M) of the EM [23-26] according to the equation,

$$SE(dB) = \mathrm{SE_R} + \mathrm{SE_A} + \mathrm{SE_M} = -10\log(P_T / P_I) \quad (4)$$

where, P_I and P_T are the power of incident and transmitted EM waves, respectively. According to Schelkunoff's theory, SE_M can be ignored for all practical purposes when the shield is thicker than the skin depth (δ) of the material. Furthermore, SE_R and SE_A can be calculated using equations, $SE_R = -10\log(1-R)$ and $SE_A = -10\log(1-A_{eff}) = -10\log(T/1-R)$

For a material, the skin depth (δ) is the distance up to which the intensity of the EM wave decreases to 1/e of its original strength. δ is related to angular frequency and relative permeability through the relation, $\delta = \sqrt{2/\sigma\omega\mu}$ ($\sigma_{ac}=\omega\varepsilon_0\varepsilon''$, see supplementary information Figure S1). Determination of the critical thickness of the shield and its variation with frequency has been shown in supporting information (Figure S2). It should be noticed that the skin depth for SWCNT is ~ 1 mm. This means that the shield should have a thickness greater than ~1 mm.

Figure 3 (c) shows the variation of SE with frequency in the 12.4-18 GHz range. The values of SE due to absorption (SE_A) are; -10.5 dB, -12.5 dB and -19.5 dB for a thickness of 1.00, 1.25 and 1.50 mm, respectively. Thus, SE_A increases with increase in thickness. On the other hand, the values of SE_R are; -2.0 dB - 4.5 dB, -4.5 dB, respectively. Therefore, the total SE for a thickness of 1.00, 1.25 and 1.50 mm are; -12.5, -17.0 and -24.0 dB, respectively. Thus, SE is mainly dominated by absorption as usually reported for such materials. It is to be noted that the value of SE for a critical thickness of 1.5 mm is greater than the limit required for commercial application (~-20 dB).

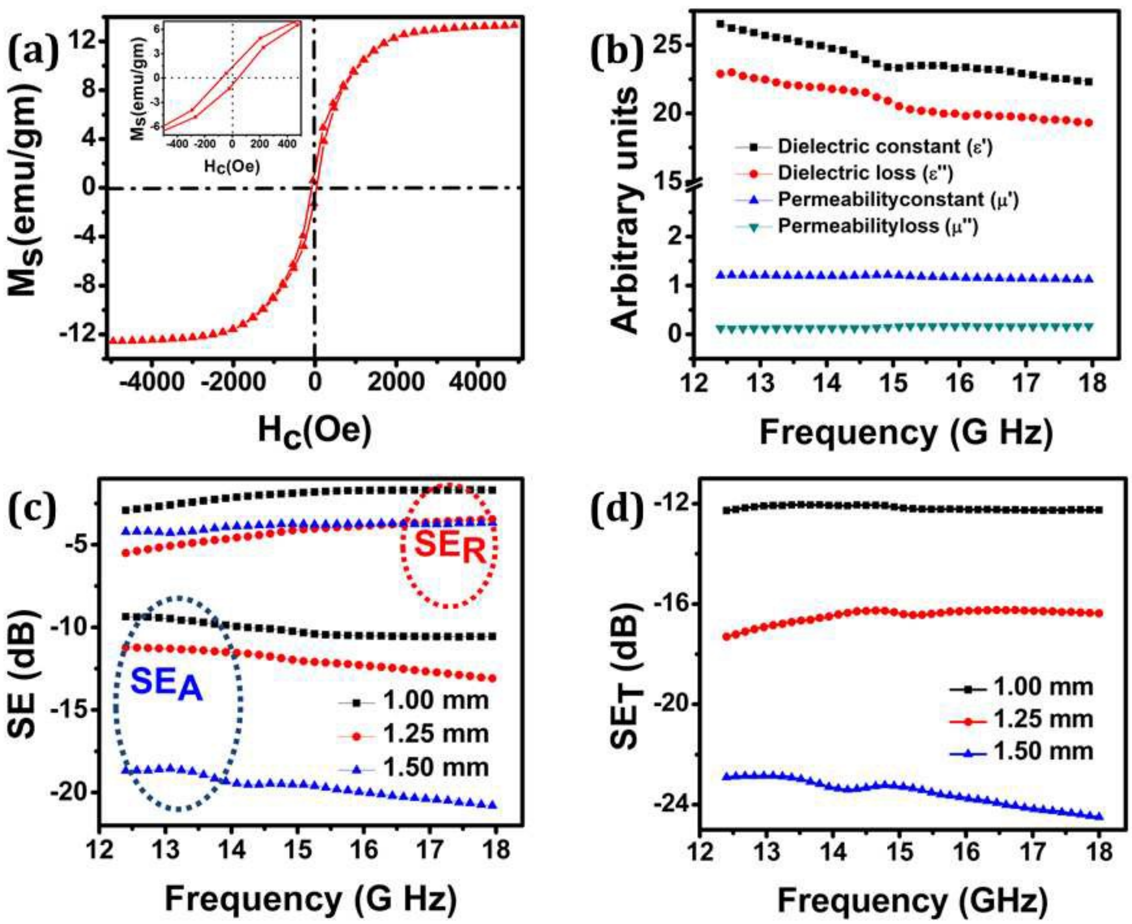

Figure 3. (a) M-H curve for Co/Ni @SWCNTs, inset of image (a) shows the retentivity and coercivity in the M-H plot of Co/Ni @SWCNTs, (b) frequency dependence of the real and imaginary parts of the complex permittivity and permeability, (c) effect of thicknesses on the shielding effectiveness (SE_A and SE_R) for composite in the frequency range of 12.4 –18 GHz and (d) total SE of Co/Ni @SWCNTs as a function of frequency

The obtained SE results can be explained in terms of electromagnetic attributes (ε^* and μ^*) of Co/Ni @SWCNTs. The complex permittivity ($\varepsilon^*=\varepsilon'-i\varepsilon''$) and complex permeability ($\mu^*=\mu'-i\mu''$) have been calculated using the experimental scattering parameters (S_{11} and S_{21}) using standard Nicholson-Ross and Weir theoretical calculations[27]. The results are shown in Figure 3 (b). In short, permittivity (ε') and permeability (μ') accounts for the amount of polarization in the material and symbolizes the storage ability of the electric and magnetic energy. The permittivity (ε'') and permeability loss (μ'') reveal the dissipated electric and magnetic energies, respectively. As shown in Figure 3 (b), the values of ε′, ε″, μ′ and μ″ lies in the range of 22.2-26.5, 19.3-22.9, 1.12-1.20 and 0.16-0.12, respectively, i.e., in the Ku-band frequency range. The observed permittivity (ε′) is due to conductivity, electronic polarization, dipole polarization as well as interfacial polarization. Similarly, dielectric losses (ε″) are result of electronic polarization and its relaxation, dipole relaxation, natural resonances and structure of the shield. Interfacial polarization in heterogeneous media is due to accumulation of charges at the interfaces and formation of dipoles. Interfaces between SWCNT, Co nanoparticles, Ni nanoparticles further contribute to the dielectric losses. The ballistic electrons in the SWCNT cannot reorient themselves fast enough when external electric field is applied. Magnetic nanoparticles of Co/Ni present onto SWCNT acts as polarization centre and contribute towards better microwave absorption. Higher aspect ratio and good conductivity of SWCNT also enhance the absorption properties. The observed permeability is due to the presence of Co/ Ni magnetic nanoparticles which act as tiny dipoles and get polarized in the presence of electromagnetic field resulting in better microwave shielding. Furthermore, eddy current due to magnetic particles and natural resonance caused by the enhanced surface anisotropy of the small sized Co/Ni nanoparticles also contribute towards better absorption. Anisotropy energy of the nanosized materials would be higher due to surface anisotropic field [28, 29]. The higher anisotropy energy also contributes in the enhancement of the microwave absorption. Several mechanism for enhanced EMI shielding and microwave absorption on CNTs based materials have been proposed earlier[30-32].

Thus, the excellent microwave absorbing performance by Co/Ni @SWCNTs is mainly attributed to the impedance matching and microwave attenuation. For a perfect absorber, $\varepsilon_r=\mu_r$ or $\varepsilon_r/\mu_r=1$. The presence of super-paramagnetic Co and Ni nanoparticles onto SWCNT helped in lowering the εr/μr ratio resulting in improved impedance matching [29]. In order to provide a visual demonstration of the microwave shielding mechanism (as discussed above), a schematic diagram has been shown in Figure 4. The above results prove that Co/Ni @SWCNTs could be potentially used as microwave shielding material.

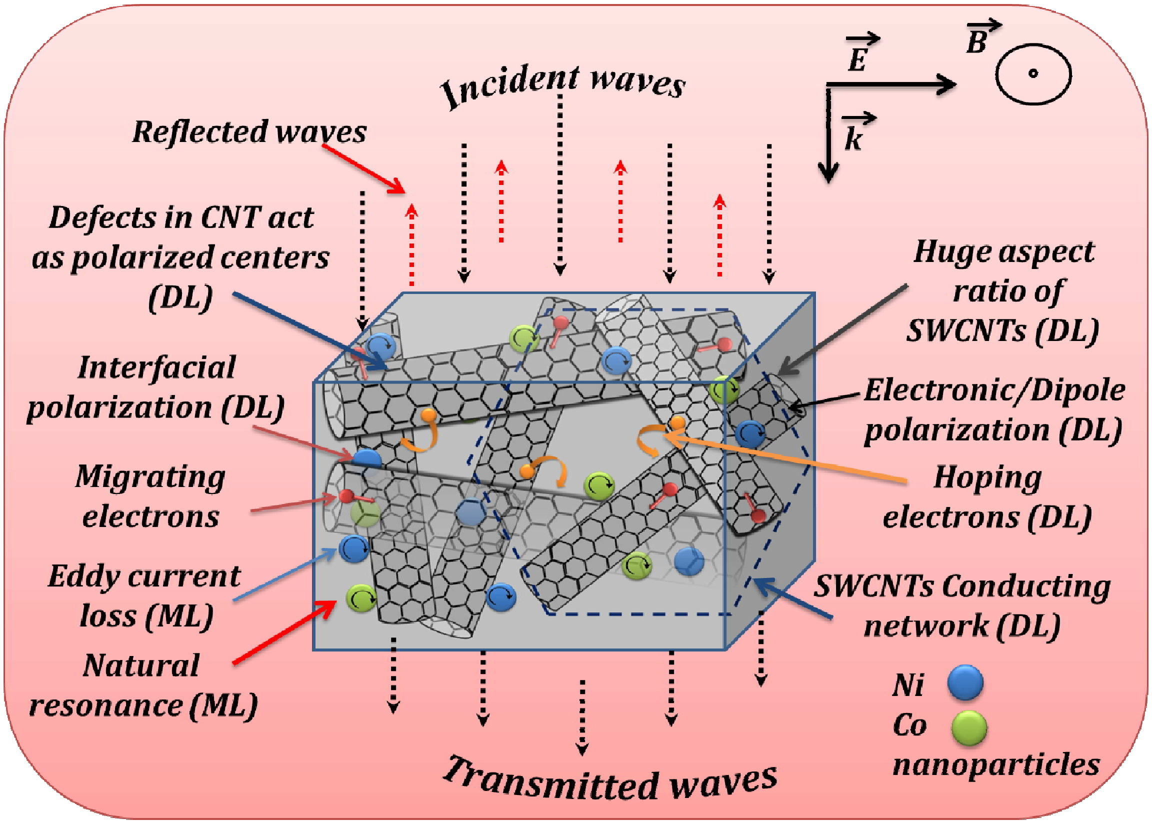

Figure 4. Schematic presentation of the interaction of EM wave with Co/Ni@ SWCNTs

4. Conclusions

Cobalt/nickel nanoparticles decorated single-walled carbon nanotubes were successfully synthesized by dc-arc discharge technique and their dielectric and magnetic performances were investigated. TEM results showed that Co/ Ni nanoparticles were attached onto SWCNTs. At 15GHz frequency, a value of 22.7 for dielectric constant 21.2 for dielectric loss, 1.2 for magnetic permeability 1.2 and 0.1 for permeability loss 0.1 was observed for Co/Ni @SWCNTs sample. At an external applied field of 5 kOe, the saturation magnetization value for the Co/Ni @SWCNTs was ~12.25 emug^{-1}. A microwave shielding effectiveness value of 24 dB for 1.5 mm thick sample in the frequency range 12.4-18 GHz was observed which is attributed to dielectric (electronic and interfacial polarization) and magnetic (eddy currents and natural resonances) losses. This new material showed microwave shielding effectiveness value greater than required for commercial applications. Thus, these nanocomposites are promising microwave shielding material in the Ku band.

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Graphical Abstract

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