

FULL PAPER

# Influence of implantation nitrogen gas on electrical properties of magnetic CNT-Fe3% and CNT-Fe5% composite

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**Graphical abstract** 



#### Abstract

The composite of CNT–Fe were made by mixing CNT and Fe powder with the variance of Fe starting from 3% and 5% weight. Then the sample is milling for 2 hours using High Energy Milling (HEM). The CNT-Fe Composite to be had done implantasion with nitrogen gas for 5 hour and 8 hour. The result of magnetic parameter of composite CNT-Fe3% and CNT-Fe5% with VSM (Vibrating Sample Magnetometer) method shows that the remanent magnetic (Mr) and saturation magnetic (Ms) increased, and the coersive magnetic (Hc) decreased with the increasing of weight percent of Fe. The result of electrical properties of composite CNT-Fe3% and CNT-Fe5% using LCR instrument indicated that conductivities value of composite CNT-Fe3% and MWCNT-Fe5% are increased with the increasing of Fe weight. The surface morphology of composite CNT-Fe3% and CNT-Fe5% was done with TEM (Transmition Electron Microscopy) with the result that Fe was had into CNT.

Keywords: Composite CNT-Fe, implantation, magnetics, conductivity, surface

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#### INTRODUCTION

Carbon nanotubes (CNTs) have two types of Single Walled Nanotubes (SWCNT), which is a graphite sheet rolls with short size, has a one-dimensional structure. Other forms of multi walled CNT is Carbon Nanotube (MWCNT) have more than a one-dimensional structure and its size is shorter by Shanov et al. (2006). Solid conductor has a conductivity depend on the frequency and temperature, solid conductor at a certain temperature can be take place to change a phase transition by Yulkifli et al. (2009) and Blanton et al. (2011). Values electric resistivity about 10<sup>-6</sup> ohm.cm for SWCNT and MWCNT while 3x10<sup>-5</sup> ohm.cm. Shows that the CNT is a conductor who has better resistivity value of metals such as Cu at room temperature. The existence of defects in the CNT will affect the conductivity of CNTs are defect structures by Li et al. (2007) and Harris (2004) and Soo Kim et al. (2008). Technology applications in the electrical properties of CNTs in electronic nanostructures, namely high frequency, field emission and sensing biology by Sup So et al. (2011) and Bandaru (2007). The growth of CNTs with nitrogen so lowering the activation energy for nucleation and layer growth by Young Kim et al. (2003). The composite of CNT-Fe result from milling and to be pressure about 7 N/m<sup>2</sup>. Then composite of CNT-Fe to be implantation with nitrogen gas so that deformation occurs between CNT-Fe composite with nitrogen gas. Implantation effect this may to defects that will improve the electrical properties. The parameter obtained have been applied in the application of carbon-based nano composite materials for sensors by Sinha et al. (2006) and Sinha et al. (2005). Purpose of this study to obtain optimu parameters

good magnetic properties, electrical and surface morphology respectively.

#### **EXPERIMENTS**

Materials used in this experiment is a powder Multi Walled Carbon Nanotube (MWCNT) brand "cheap tube" purity more than 95%, and iron powder (Fe) Aldrich product which has a purity of 99.9%. Fe powder and MWCNT weighed with the following composition weight: MWCNT (97%)-Fe (3%) and MWCNT (95%)-Fe (5%). The total weight of each powder mixture is 10 gr.

The powder mixture is process milling for 2 hours with a technique High Energy Milling (HEM), brand SPEX CertiPrep 8000M Mixer/Mill. Mix powder milling processes were characterized by the electrical properties of LCR-meter and magnetic properties by VSM (Vibrating Sample Magnetizer). CNT-Fe composite to be implantation with nitrogen gas for 5 hours and 8 hours. The results to be observated on CNT-Fe composites using the VSM (Vibrating Sample Magnetizer) to determine magnetic properties, electrical properties with LCR-Meter and the surface of morphology by TEM (Transmission Electron Microscopy) in the composite material.

## **RESULTS AND DISCUSSION**

Figure 1a. shows relationship between the magnetic moment versus the field strength on CNT-Fe material with Fe concentration variation before the implant by Purwanto et al.(2014). Figure 1b and Figure 1c, shows relationship between magnetic moment versus the

field strength on CNT-Fe material with Fe concentration variations after implant. The Parameter magnetic of CNT-Fe composite before and after implant show at Table 1 and Table 2.



**Figure 1** (a). The relationship between coersive field versus moment magnetic of CNT-Fe before implant (Figure 1a, data from the reference to be has been published by P.Purwanto, et al, 2014); (b) CNT-Fe3% after implant; (c) CNT-Fe5% after implant.

Saturated magnetic moment (Ms) which indicates the ability of a material to receive the magnetic field strength increases with increasing Fe weight, except for time the implant 5 hours (CNT-Fe5%). The saturated magnetic moment (Ms) occurred irregularities and Fe mixture CNT time miling process. While the remanent magnetic moment (Mr) which shows the nature of the material is still a magnet properties when the absence of a magnetic field. With the rising Mr indicates that composite of CNT-Fe is have to properties of ferromagnetic. In Figure 1b, shows that the remanent magnetic moment to rise with increasing of Fe weight. Rising Mr showed that composite of CNT-Fewill be properties of ferromagnetic.

 Table 1
 Parameter magnetic CNT-Fe3%, satureted moment (Ms), remanent moment (Mr) and coersive field (Hc).

Time implant	Ms ( emu/g )	Mr ( emu/g )	Hc ( Oe )
0 hour	6.2	1.30	168
5 hour	12.7	2.74	665
8 hour	11.2	2.12	648

 
 Table 2 Parameter magnetic CNT-Fe5%, satureted moment (Ms), remanent moment (Mr) and coersive field (Hc).

Time implant	Ms ( emu/g )	Mr(emu/g)	Hc(Oe)
0 hour	7.9	1.70	136
5 hour	11.8	0.75	134
8 hour	20.4	1.05	161

Table 1 and Table 2, show that coercive field (Hc) which indicate whether the material is non isotropy or isotropy properties if to be related to materials that are strong or weak when subjected to the magnetic field. From the research that a coercive field (Hc) down to the addition of Fe weight after implantation with nitrogen gas. While coercive field (Hc) after implantation is increase with time the implant. Effect of implantation with nitrogen gas can increase the field strength coercive. Increasing coercive field is an interaction between the composite of CNT-Fe with nitrogen gas that to be become ions, so that ions of nitrogen gas into the composite of CNT-Fe.

The composite CNT-Fe indicate, that moment for a long time the implant, the results shown for the composite CNT-Fe5 (MS-5) is higher than the CNT-Fe3% (MS-3). While the length of time the implant 5 hours CNT-Fe5% moment (MS-5) dropped significantly. Coercive field is increased with increasing time the implant, while the coercive field at CNT-Fe3% higher than the CNT-Fe5% shown in Figure 1b and Figure 1c. Remanent magnetic moment (Mr) composite of CNT-Fe is irregular against of time the implant.

The electrical conductivity of CNT-Fe composite before and after the implant shown in Figure 2. Conductivity of CNT-Fe3% lower than CNT-Fe5% with increasing of Fe weight and conductivity curve CNT-Fe3% and CNT- Fe5% with increasing frequency. Conductivity of composite CNT-Fe3% and CNT-Fe5% after implant is decreased with long time the implant and curve of relative flat with increasing of frequency.

Conductivity of composite CNT-Fe being dropped with it take place interaction between nitrogen ions with composite of CNT-Fe, so that forming a layer that resist the movement of electrons in a CNT. The composite of CNT-Fe5% conductivity have value is higher than the value of the conductivity of CNT-Fe3% and higher than before the implant with nitrogen gas. According by Padma et al.(2006), a material defect when to be began of heat treatment, radiation and concentration difference, the conductivity will inceased with many defects that occur in the composite material. From these results CNT-Fe composite conductivity decrease with time the implants and the implant of nitrogen gas. Effect of implants with nitrogen gas at CNT-Fe composite materials form a defect is irregular. Meanwhile, according to Chandra defect that Frenkel defect or a defect of Shoctky. Calculation of the value of conductivity at CNT-Fe composite using models by Lu et al. (2008), with using the equation:

$$\sigma(\omega) = \sigma_{0+} A.\omega^{.s} \tag{1}$$

where:  $\sigma(\omega)$  is the AC conductivity (S/cm),  $\sigma_0$  is DC conductivity when  $\omega = 2\pi$ .f=0 (conductivity does not depend on frequency), A is the pre-exponential factor and s is exponential factor (0 < s < 1). The calculation result of conductivity of CNT-Fe before and after the implant with nitrogen gas shown in Table 3 and Table 4. The conductivity of a composite of CNT-Fe will increase and decrease where exist many factors taken into in the experiments that have been carried out. According by Stphen Hall.(2004), the conductivity depends on many factors such as interstitials, ion size, temperature, crystall structure, composition and phase change. Padma Kumar mentioned factors explain the defects in the crystal will cause mobilization of ions in the crystal and can increase the conductivity of the material by Padma et al.(2006). Deformation processes and the entry into the CNT particles Fe which will greatly affect the electrical properties of the composite CNT-Fe. The increase in the electrical properties of materials due to crystal defects is highly expected to occur in any area of the crystal so that the movement of electrons easily move with a small activation energy.Observation by Ionescu et al.(2012), the result that the nitrogen influence the growth rate, morphology, size and structure of nanotube, so that electrical resistance increase with temperature and pressure as nitrogen concentration increase inside the tube.



Figure 2 (a) Conductivity CNT-Fe3% and CNT-Fe5% before implant; (b) Conductivity CNT-Fe3% after implant (c) Conductivity CNT-Fe5% after implant

Table 3 Conductivity composite         CNT-Fe before the implant				
Name Sample	$\sigma_{\rm o}$ ( S/cm )			
CNT-Fe3%	0.87×10 <sup>-1</sup>			
CNT-Fe5%	2.00×10 <sup>-1</sup>			

Table 4 Conductivity composite CNT-Fe after the implant				
Name Sample	Time implant	$\sigma_{\rm o}($ S/cm $)$		
	5 hour	4.79×10 <sup>-5</sup>		
CINT-Fe3%	8 hour	7.59×10 <sup>-8</sup>		
	5 hour	2.37×10 <sup>-3</sup>		
CN1-F63%	8 hour	8.45×10 <sup>-4</sup>		

Figure 3 indicate surface of the composite of CNT-Fe3% and CNT-Fe5% by transmission electron microscopy (TEM) with a magnification of 40.000 X. From the observation with TEM looked Fe into the black CNT round as well for CNT-Fe5%. In this experiments, the amount of Fe into CNT can not be counted. To determine the amount of Fe is entered into the CNT performed with conductivity analysis of each CNT-Fe composite. The amount of Fe which entered into CNT indicated by the conductivity curve. Figure 3, so that Fe into composite of CNT-Fe5% with shows the conductivity value is higher than the CNT-Fe3%.



**Figure 3** Surface of CNT-Fe composite with a transmission electron microscope (a) Surface CNT-Fe3%; (b) Surface CNT-Fe5%.

## CONCLUSION

Results of measurement of magnetic properties with the method VSM (Vibrating Sample Magnetometer) indicates that the moment magnetic remanent (Mr), saturated magnetic moment (Ms) increased, where as the coercive field (Hc) decreased in proportion to the increase in the concentration of Fe with persent weight. After the implant treatment with nitrogen gas showed that the magnetic moments of magnetic saturation and remanent was down, except

CNT-Fe3%. The coersive magnetic (Hc) decreased with the increasing of weight percent of Fe.

Results of CNT-Fe conductivity measurement with LCR measuring instrument, indicates that the value of conductivity increases proportional to the increase in the concentration of Fe weight. Conductivity CNT-Fe3% and CNT-Fe5% after implant with nitrogen gas to decrease with the length of time the implant. From the observation with TEM looked Fe entered into CNT black tube and outside round so well of CNT.

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