

Synthesis and Properties of Ni-CNT Nanocomposites Using Electrical Explosion of Wire in Different Conditions

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Abstract Ni-CNT nanocomposites were synthesized via the electrical explosion of wire (EEW) in acetone and deionized (DI) water liquid conditions with different CNT compositions. The change in the shape and properties of the Ni-CNT nanopowders were determined based on the type of fluids and CNT compositions. In every case, the Ni nanopowder had a spherical shape and the CNT powder had a tube shape. However, the Ni-CNT nanopowders obtained in DI water exhibited irregular shapes due to the oxidation of Ni. Phase analysis also revealed the existence of nickel oxide when using DI water, as well as some unknown peaks with acetone, which may form due to the metastable phase of Ni. Magnetic properties were investigated using a Vibrating Sample Magnetometer (VSM) for all cases. Nanopowders prepared in DI water conditions had better magnetic properties than those in acetone, as evidenced by the simultaneous formation of super paramagnetic NiO peaks and ferromagnetic Ni peaks. The DI water (Ni:CNT = 1:0.3) sample revealed better magnetic results than the DI water (Ni-CNT = 1:0.5) because it had less CNT contents.

Key words Ni nanocomposite, electrical explosion of wire, carbon nanotube, magnetic property.

1. Introduction

Nanosized nickel-carbon (Ni-C) composites received great attention for hydrothermal gasification of organic compounds,^{1,2)} application in electrochemical devices as sensors³⁾ and electrodes,⁴⁾ or hydrogen storage.⁵⁻⁹⁾ Several types of carbon materials have been used to prepare the Ni-C composites such as activated carbons, graphite, carbon black, etc.^{1,2,6,7,10)} Recently, new carbon forms such as carbon nanotubes (CNTs) and magnetically separable carbon materials have been synthesized.^{3,4,8,11-13)} The CNTs are one of the most exciting new materials, which possess excellent mechanical properties as well as thermal and electrical characteristics. Also it shows less magnetic properties, so by decreasing the CNT compositions in Ni-CNTs nanopowders, we can get some different results.

A new method, the Electrical Explosion of Wires (EEW), can be used to fabricate Ni-CNT nanocomposites effectively.

The EEW is a single-step physical synthesis technique that can be finished within a second, because it does not require a precursor or additional heating steps. This method involves the use of a pulsed electric current to heat wire, synthesizing high-purity nanopowders without a surfactant. With the EEW method, the Ni-CNTs was fabricated not only in the air, but also in a liquid media. The explosion of wire in liquid have a potential to prepare nanocomposites in one-step technique, in which the nanopowders was simultaneously made and dispersed into the base liquid, resulting in reduction of the agglomeration of the nanopowders and therefore increasing in the stability of the suspension. In this process, a metal wire is exploded under a high pulsed electric current.¹⁴⁾

Here acetone and deionized (DI) water were used as the ambient liquids. As acetone is a volatile, flammable liquid and DI water is a stationary like liquid, that's why this two types of liquids were chosen as ambient conditions to compare with each other. DI water don't has many of its

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impurities through distillation. In addition, since the acetone contains many carbon components, it is expected that metal carbides can be formed during the explosion, so the carbide formation process could be compared with the DI water condition. Here Ni:CNT compositions were 1:0.5 and 1:0.3 in weight.

2. Experimental Procedure

2.1. Materials and synthesis

To fabricate Ni-CNT nanocomposite, in this work, high purity CNTs was used and Ni nanopowders were fabricated by EEW in acetone and DI water. The EEW system was a simple RLC circuit, as shown in Fig. 1. The capacitance C was proportional to the applied voltage, and the resistance R and inductance L were determined by the material's properties and dimensions of the wire.

Firstly, the CNTs were mixed well in 500 mL of acetone and DI water, respectively, by stirring process at room temperature for 2 h. Then Ni wire with diameter 0.25 mm in diameter and 30 mm in length was placed between the two electrodes and exploded in the slurry of the carbon nanotubes. A 30 μF capacitor was charged up to 3 kV and then released through the wire when the spark gap switch was

closed. Following that way, a high-density current was passed through the wire and deposited the electric energy on it due to its finite resistance. Under the effects of high-density current, the wire between two electrodes was melted, vaporized, turned into plasma and then expands into the ambient liquid. The Ni vapor was immediately cooled down rapidly because of the interaction with the liquid solution resulting in nanosized powders were formed and dispersed in the acetone or DI water solutions. The Ni content was estimated by the times of explosion and the dimension of the Ni wire section according to the equation: $m = \rho \cdot V$, where m is the weight of Ni wire and V is the volume of the Ni wire was used for one explosion shot. Synthesis conditions are listed in Table 1.

2.2. Characterization methods

In all cases, after explosion, the powders were isolated from the suspension via centrifugation and dried under vacuum for 5 h. Morphology of the Ni-CNT nanopowders was evaluated by field emission scanning electron microscopy (FE-SEM, JEOL JSM-6500F). A small amount of the Ni-CNTs was taken out and collected for phase analysis by X-ray diffractometer (XRD). The XRD pattern was recorded from 20° to 90° (2θ). A magnetic measurement for the Ni-CNTs was performed in the room temperature by using a

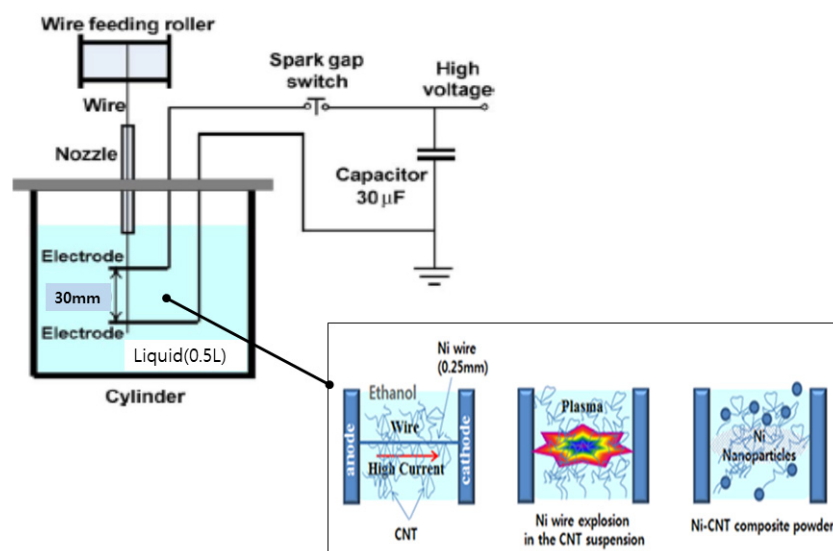


Fig. 1. Schematic diagram of experimental setup for wire explosion process in liquid.

Table 1. Conditions used for the synthesis of Ni-CNT nanocomposites.

Capacitance	Charging voltage	Wire diameter : Nickel	Wire length of one explosion	Ambient liquid
30 μF	3 kV	$\Phi 0.25$ mm	30 mm	Acetone and DI water

Vibrating Sample Magnetometer (VSM).

3. Results and Discussion

3.1. Phase analysis

Fig. 2 presents the XRD patterns of the nanopowders collected from fluid by centrifuging of the Ni-CNT nanofluids. The X-ray diffraction data of the samples was recorded for the entire angular range of 20–90 °C. Carbon hexagonal phase is extremely oriented along (002) for all conditions with very low intensity. The relative intensities of Ni (FCC) reflections are in accordance with the JCPDF files # 04-0850. Here Ni FCC phase along (111), (200), (220) are noticed to present in all conditions. Also the powders prepared in acetone and DI water showed some unassigned peaks, because carbon and oxygen atoms in these two liquids are thought to have been incorporated in the nickel crystal structure to form metastable-phase.¹⁵⁾

In case of acetone for both CNT compositions, the shifting tendency of Ni peaks is due to the diffusion of carbon atoms into the Ni-lattice which leads to form Ni₃C solid solution alloy at high plasma condition.¹⁶⁾ These carbon atoms come actually from the carbon chain of the acetone itself. No Ni₃C peaks was detected in the DI water condition.

However, the powder produced in deionized water showed a mixed phase with pure nickel and nickel oxide (NiO). The peak intensity of pure nickel is much stronger than that of nickel oxide. This NiO formed because Ni element reacted with oxygen diluting in water when the Ni vapor was condensed. The formation of the nickel oxide in

the EEW in the DI water is almost the same as the formation of Cu oxide in the DI water.¹⁷⁾ Quantitative analysis of the formation of the Ni carbide and Ni oxide during the explosion in various liquids is very interesting, so it will be carried out in future studies.

3.2. Morphology and size of nanopowders

Fig. 3 illustrates the morphology and particles size of Ni-CNT nanopowders prepared in acetone and DI water for 1:0.5 and 1:0.3 compositions. From the images, it was observed that the Ni particles had nearly spherical shape. The particles size included big and small particles. The smaller ones are less than 100 nm, but the larger ones are almost 1 μm. The small particles seem to aggregate into big clusters. And also the unbroken tube shaped CNT particles could be found in all images easily. But in case of DI water (1:0.5), the spherical Ni nanoparticles was in quite some irregular shapes (inset image). This shape is because of oxidation of the nickel element. In water, there is an amount of free oxygen dissolving and oxygen in the water molecule. In the very high temperature of plasma state, the nickel element was oxidized in case of DI water and formed oxide phase, which was already revealed in XRD analysis.

From the EDS profile of Fig. 4, we could see the presence of oxide phase in both compositions for DI water liquid conditions which was already revealed in the XRD results. And in the EDS results, in case of the nanopowder prepared in acetone there was no oxidation. Table 2 shows the atomic and weight percentage of Ni, C and O in the as-prepared nanopowders.

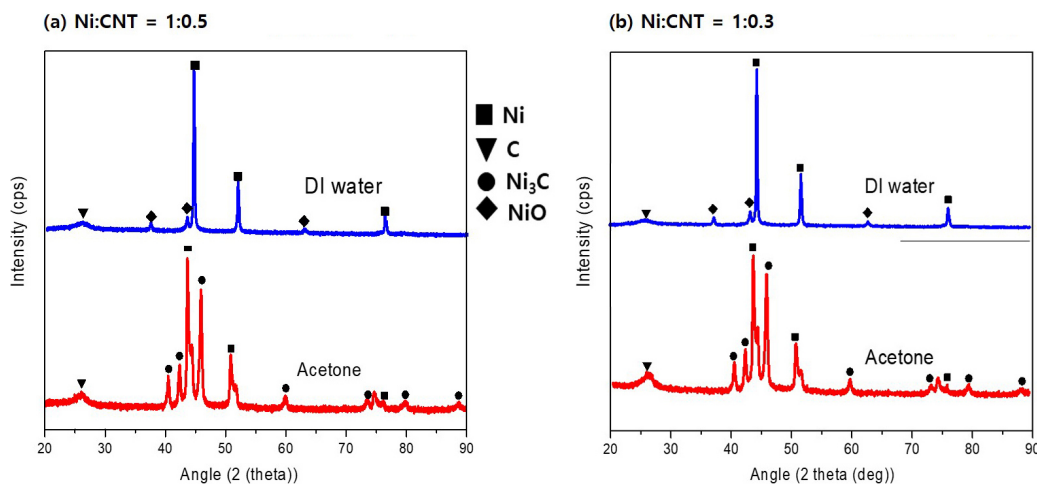


Fig. 2. XRD patterns of Ni-CNT nanopowders produced via EEW in acetone and DI water (a) 1:0.5, (b) 1:0.3.

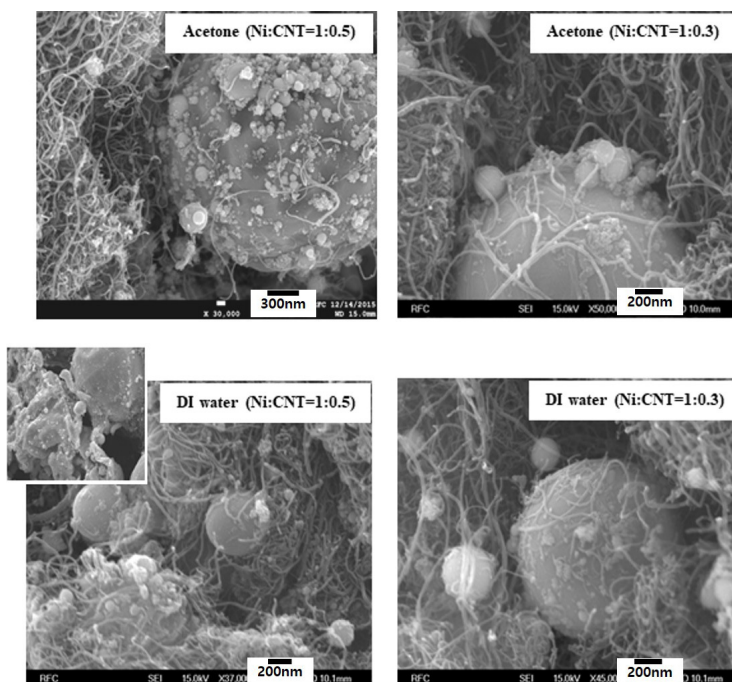


Fig. 3. FE-SEM images of Ni-CNT nanopowders produced via EEW in acetone and DI water for 1:0.5 and 1:0.3.

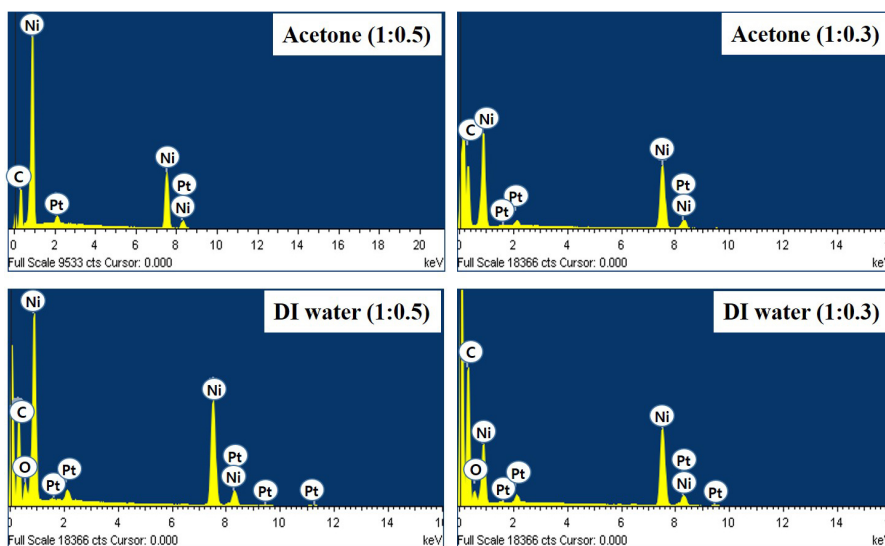


Fig. 4. EDS profile of Ni-CNT nanopowders produced via EEW in acetone and DI water for 1:0.5 and 1:0.3.

Table 2. Percentage of Ni and C in Ni-CNT composites.

Element	(%)	Acetone (1:0.5)	Acetone (1:0.3)	DI water (1:0.5)	DI water (1:0.3)
C K	Weight (%)	41.68	65.68	59.89	71.31
	Atomic (%)	77.74	90.34	81.17	87.18
Ni K	Weight (%)	58.32	34.32	29.69	20.23
	Atomic (%)	22.26	9.66	8.23	5.02
O K	Weight (%)	-	-	10.42	8.45
	Atomic (%)	-	-	10.60	7.76
Totals	-	100.00	100.00	100.00	100.00

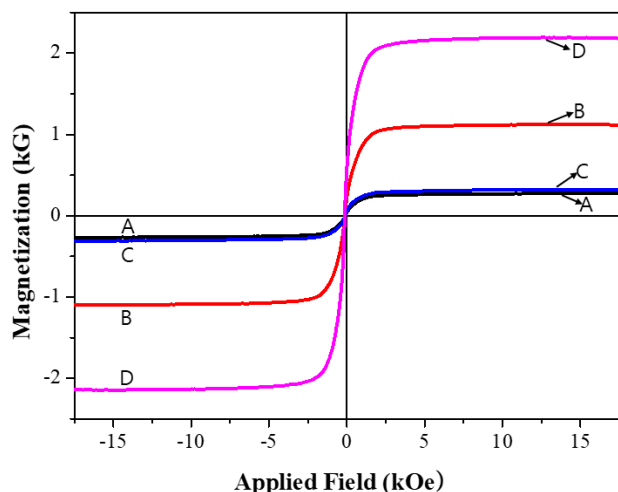


Fig. 5. Hysteresis loops of the Ni-CNT nanopowders measured at room temperature - (A) acetone (1:0.5), (B) DI water (1:0.5), (C) acetone (1:0.3) and (D) DI water (1:0.3).

Table 3. Coercivity and saturation magnetization of the Ni-CNT nanopowders measured at room temperature.

Elements	Applied field, H (Oe)	Magnetization, M (emu/g)
Acetone (1:0.5)	83.768	0.3657
DI water (1:0.5)	132.05	1.7821
Acetone (1:0.3)	97.170	0.5076
DI water (1:0.3)	126.85	3.4506

3.3. Magnetic properties

Vibrating sample magnetometer (VSM) test was used to determine the magnetic properties of nanopowder. It was investigated at room temperature with an applied field $-20,000 \text{ Oe} \leq H \leq 20,000 \text{ Oe}$. Fig. 5 shows the magnetic hysteresis loops for two different CNT compositions of Ni-CNT nanopowders produced in acetone and DI water. The values of saturation induction and coercivity were listed in Table 3.

Here the as-prepared powders in acetone showed the lowest magnetization value in both compositions than that in the DI water. The presence of so many peaks of Nickel-carbide with a complex structure in the powder synthesized in the acetone which already revealed in XRD results, were the main cause of this lower magnetization value of the acetone. From the XRD results of the Ni-CNT nanopowders prepared in the DI water, we could see ferromagnetic Ni peaks and some very small super paramagnetic NiO peaks with no nickel-carbide peaks at all. So that, it showed the

best magnetization value than the acetone.

Also it could be said undoubtedly that, the nanopowders having lower CNT compositions showed better magnetic results than the nanopowders having higher CNT compositions for both liquid conditions due to the variations of CNT content.

4. Conclusion

Ni-CNT nanopowders were fabricated by EEW in acetone and DI water for different CNT compositions. The XRD diffractogram showed the same peaks and peaks position for same liquids in different compositions. The morphology analysis was also quite similar for both liquid conditions of different CNT compositions. The magnetic results showed some different results. By decreasing CNT compositions, the magnetization value became higher than the higher CNT compositions nanopowders for both liquid conditions due to having lower CNT content. And because of formation of Ni_3C peaks, the nanopowders synthesized in acetone showed smaller magnetization value than DI water.

So after that we may conclude by considering that, among DI water and acetone, DI water is the best ambient liquid to prepare Ni-CNT nanopowder by EEW. And DI water (1:0.3) is good to get better magnetic results than DI water (1:0.5).

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