# Preparation of uniformly dispersed iron nanoparticles and growth of carbon nanotube

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**Abstract**

*We studied the growth characteristics of carbon nanotubes which was grown from uniformly dispersed iron nanoparticles prepared from iron-acetate [Fe(Ⅱ)$(CH_3COO)_2$]. The density of CNT was controlled from precursor concentrations. We also investigated the field emission properties of CNTs. We found that the optimization of CNT density is an important factor for field emission properties.*

## 1. Introduction

Carbon nanotube (CNT) has generated a great deal of interest due to extraordinary mechanical and electronic properties, and also many potential applications. One potential application is as a field emission electron source. CNTs have advantages as field emitters by the properties such as a high aspect ratio, chemical stability, high thermal conductivity, and high mechanical strength.

The development of uniform nanometer sized catalyst particles has been intensively searched because of their important role as a seed for CNTs growth. The synthesis of well-dispersed nanoparticles with size ranges from 2 to 10 nm enables the synthesis of thinner CNTs at lower temperature.

So far, It was very difficult to disperse particles uniformly on the substrate. In this paper, we get well dispersed substrate with catalyst powder using by spin coating method and we also could control density of CNTs for field emission by simple experimental techniques

## 2. Experimental and Results

We used iron-acetate[Fe(Ⅱ)$(CH_3COO)_2$] as a catalyst precursor. Iron-acetate was dissolved in special solvent mixture having enough solubility and low volatility. The glass substrate was coated with catalyst solution and quickly dried to avoid recrystallization of catalyst. Al/Cr layer were deposited on glass substrates before the coating of catalyst precursor solution. Recrystallization may result larger particle size. A solution coated substrate was annealed at 350-400℃ in air to organic compound. Iron oxide particles were left on the substrate and it was confirmed from thermal gravimetric analysis (TGA) and X-ray photoelectron spectroscopy (XPS) method. Nitrogen ($N_2$) gas was then introduced while the chamber was gradually heated to the growth temperature of 550°C by infrared lamps through a quartz window, and the CNTs were grown. As soon as the temperature was reached to 550°C, a gas mixture of CO (0.08slm) and $H_2$ (1.27slm) was fed into the chamber for the growth of CNTs for 40min.

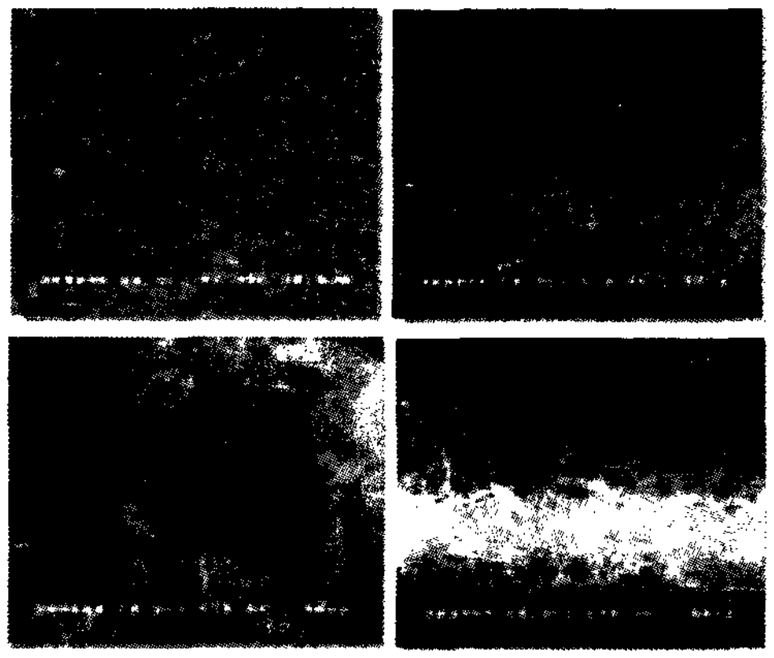

**Figure 1 CNTs grown from catalyst .solution without the control of drying process**

When the dip coating method was applied without the control of drying process, uniformity was poor by the recrystallization and coalescence of catalyst particles. Figure 1 shows the SEM images of CNTs grown by thermal CVD at 650℃ from the iron

nanoparticles coated without any control of drying process. The solution was also coated by spin coating method with controlled drying process. By changing spin speed of spin coater, the thickness of solution on substrate was controlled. Figure 2 shows the changes of CNT density by changing spin speed and more uniform distribution of CNTs.

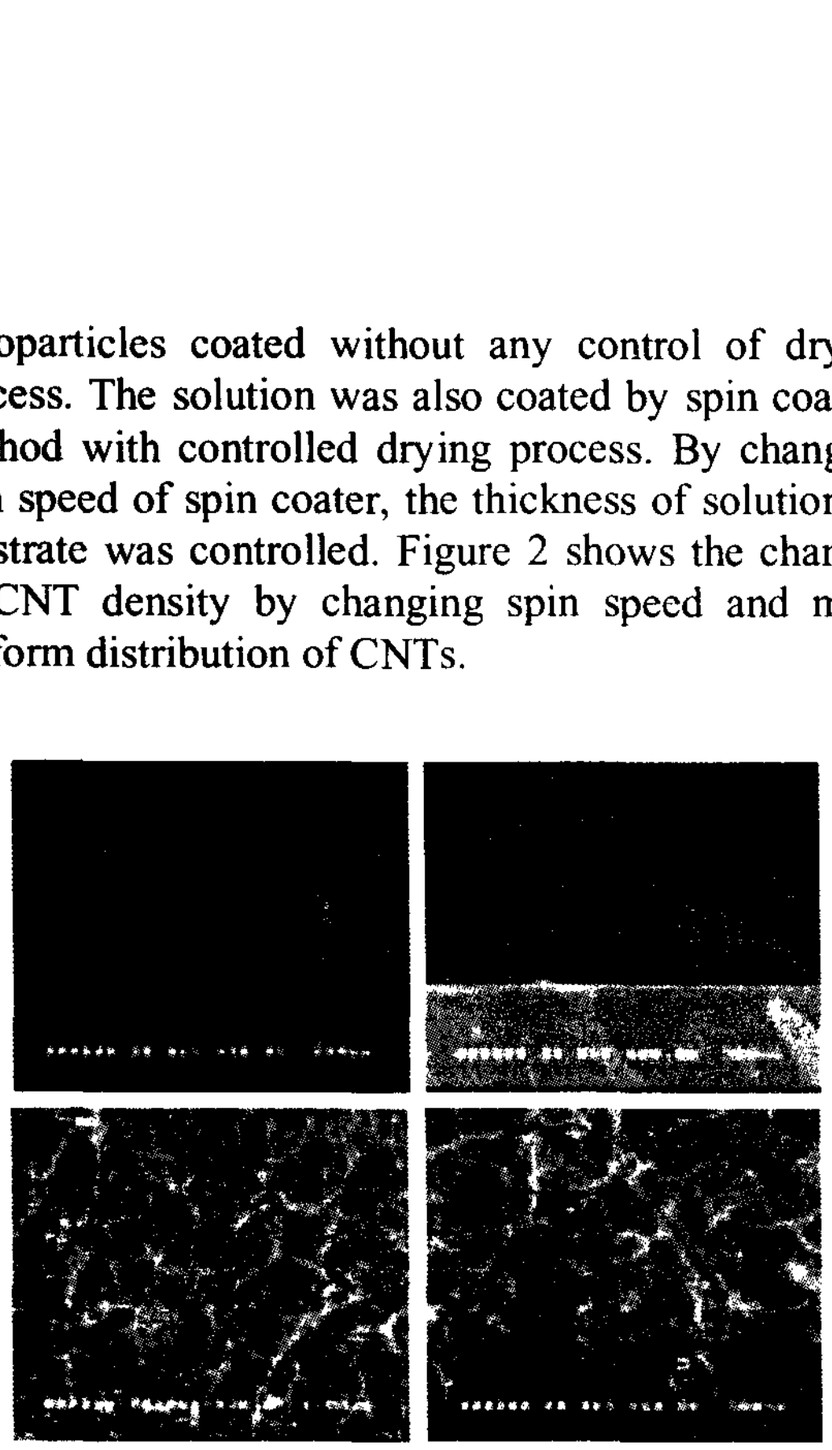

**Figure 2 CNTs grown from different spin speed during catalyst precursor coating process.**

**Top: 800 rpm, Bottom: 400 rpm**

Figure 3 shows the CNTs grown from different catalyst precursor conditions. The concentration was changed 20mM, 40mM, and 60mM and spin speed was also varied from 400rpm to 800rpm. It is reasonable to consider that the surface concentration of catalyst on substrate was increased as the concentration was increased. Moreover it will be same for the spin speed. Decreased spin speed will also increase the surface density of catalyst. As a result, it is reasonable that the surface concentration of catalyst is increased as an order of samples coated with the condition of 20mM with 800rpm, 40mM with 800rpm, 60mM with 800rpm, and 60mM with 400rpm. As the surface concentration of liquid catalyst increases, it is found that the density of CNTs was also increased. And growth rate was also increased. When coating and drying sequence was done for 3 times to increase the surface catalyst concentration, CNT length was increased about 1.5 times.

FE characteristics of CNTs were measured in a diode configuration in a vacuum of ~$3x10^{-6}$ torr. The SUS was used for the anode. The cathode-to-anode gap of 250㎛ was kept by glass spacers

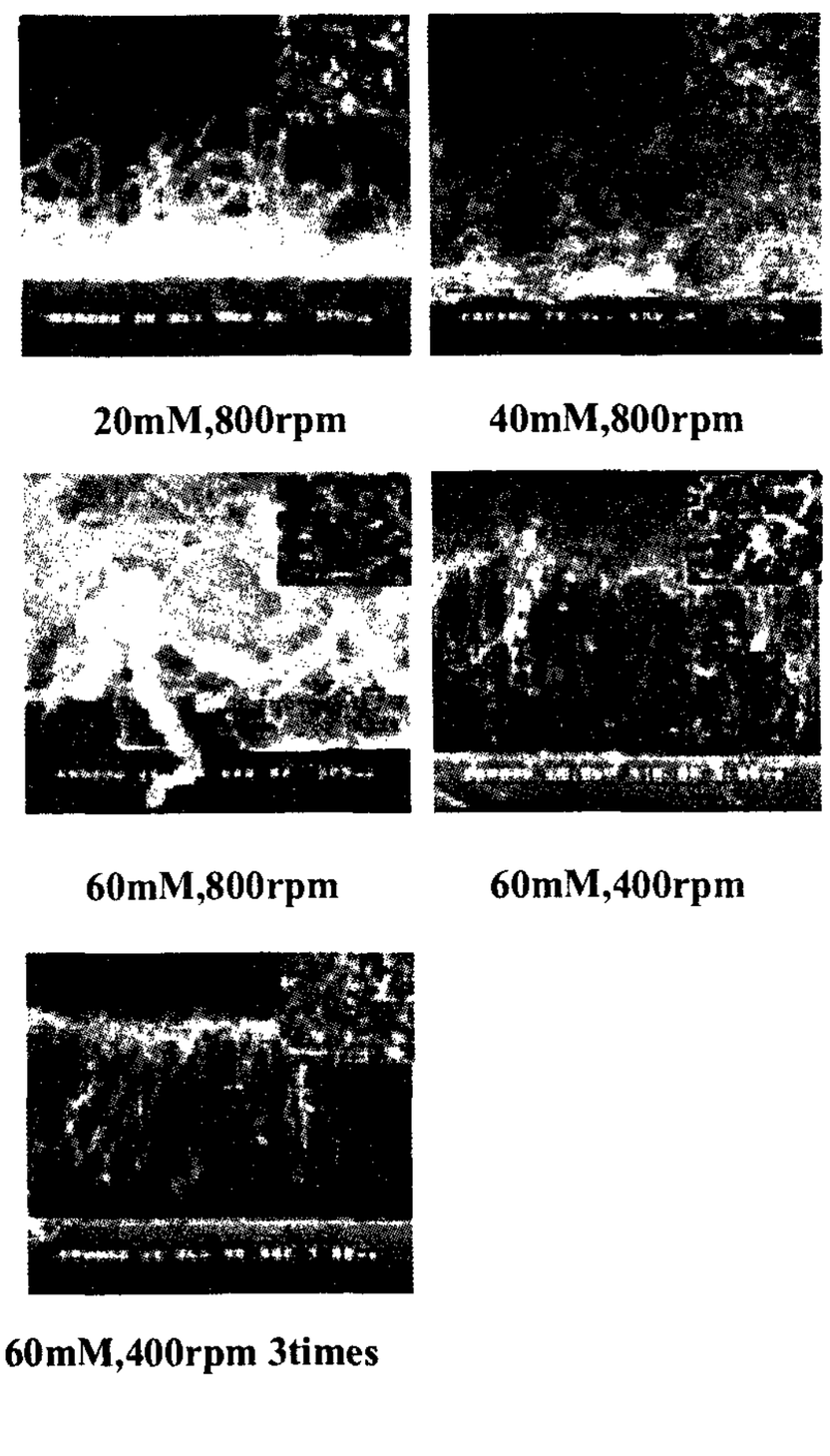


**Figure 3 CNT film configuration changes with different solution and coating conditions**

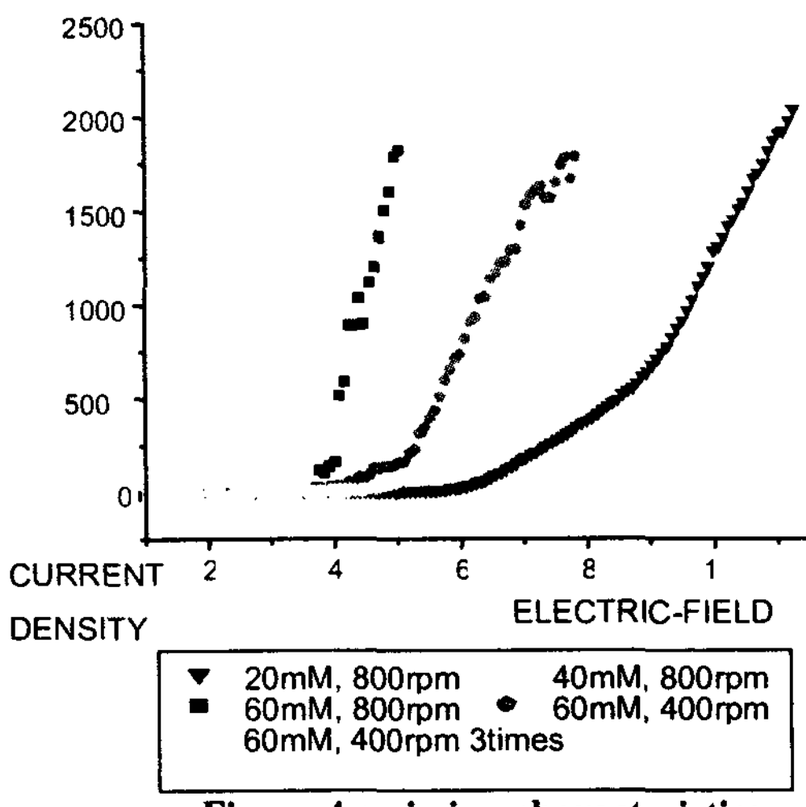


**Figure 4 emission characteristics**

Figure 4 shows the field emission characteristics of CNTs grown on the glass substrate from different catalyst surface concentrations. As we expected, there is a critical concentration and spin speed, 60mM and 800rpm respectively. The CNTs grown from lowest and highest surface catalyst concentration showed poorer field emission characteristics. It is thought that the combination of electrical field screening effect and field enhancement factor of electron emitter made optimum emitter geometry. When the catalyst concentration was too low, field enhancement factor of CNT was low resulting higher operating voltage. And when the catalyst concentration was too high, the electrical field screening effect increased operating voltage. Therefore, appropriate density of CNTs with high aspect ratio has to be designed for better electron emitting property.

## 3. Conclusion

We studied growth of CNTs from density controlled catalyst. We used spin-coating method and controlled drying (quick drying process) process to precisely control the catalyst precursor surface concentrations and to uniformly disperse iron-acetate particles on a substrate.

Finally, we found that FE characteristics varied with different concentrations, and optimum surface catalyst concentration existed.

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