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## Hybrid Double Wall Nanotube of Conducting Polymer and Magnetic Nickel

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*We synthesized hybrid double wall nanotubes of conducting polymer nanotubes enveloped by magnetic nickel (Ni) nanotubes. Nanotubes of conducting polymers such as polypyrrole (PPy) and polythiophene (PT) were synthesized in the nanoporous of alumina template through electrochemical polymerization method. Magnetic Ni nanotubes were fabricated by electrochemical deposition outside the wall of the conducting polymer nanotubes. To discern the formation of the hybrid nano-systems, we performed SEM experiments. The structural properties of the hybrid double wall nanotubes were examined by using X-ray diffraction (XRD) experiments. The ferromagnetic nature of Ni nanotubes was confirmed through the magnetic coercivity by using superconducting quantum interference device (SQUID) experiments.*

**Keywords:** nanotube; nickel; polypyrrole; polythiophene

### 1. INTRODUCTION

Nanostructured magnetic materials have attracted many attentions because of potential applications in magnetic recording media, sensors, spintronics, and nanodevices [1,2]. There have been different methods to fabricate nanostructured magnetic and electrical materials. The

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nanoporous template method has been used for the synthesis of inorganic or organic nanotube or nanowire materials, because it provides a well-defined nanoscale frame [3]. Although nanostructured magnetic materials have been fabricated by using the template method [4], the synthesis and characterization of hybrid double wall nanotubes of inorganic magnetic metals and conducting polymer have not been reported.

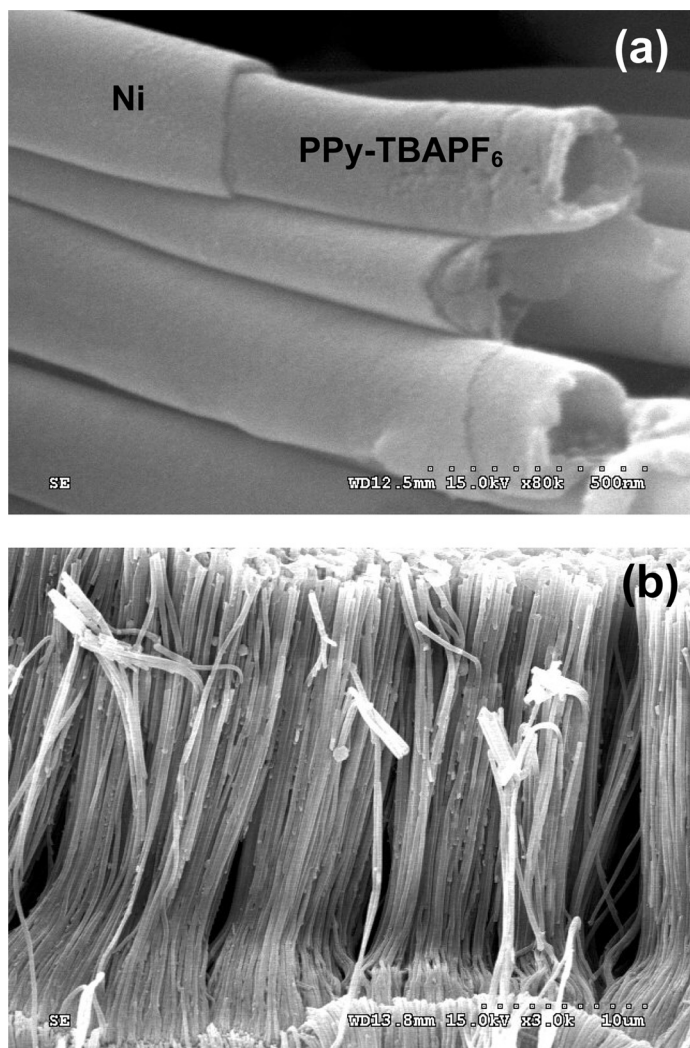
In this study, we report the synthesis and characterization of the hybrid double wall nanotubes of conducting polymer enveloped by magnetic Ni. Nanotubes of conducting polypyrrole (PPy) and polythiophene (PT) were synthesized in the nanoporous of alumina ( $\text{Al}_2\text{O}_3$ ) template through electrochemical polymerization method [3]. Magnetic Ni nanotubes were fabricated by electrochemical deposition outside the wall of the conducting PPy or PT nanotubes [4]. We observed the ferromagnetic hysteresis with the coercivity of 65 G for Ni nanotubes.

## 2. EXPERIMENTAL

Conducting PPy and PT nanotubes were synthesized through electrochemical polymerization method by using  $\text{Al}_2\text{O}_3$  nanoporous membranes (Whatman Co.) with pore diameter of 100 ~ 200 nm. The electrolyte consisted of solvent, dopant, and monomers. Thiophene and pyrrole monomers ( $\geq 99\%$  purified materials) were purchased from Aldrich. Tetrabutylammonium hexafluorophosphate ( $\text{TBAPF}_6$ ) was used as dopant, and acetonitrile ( $\text{CH}_3\text{CN}$ ) was used as solvent. The solution of electrochemical deposition for the synthesis of magnetic Ni nanotubes was composed of  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$  (270 g/l),  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (40 g/l), and  $\text{H}_3\text{BO}_3$  (40 g/l) buffered at pH 3.5 with a potential at  $-1.0\text{ V}$  ( $\text{Ag}/\text{AgCl}$ ). After polymerization of PPy or PT samples, the HF or NaOH solution was used to remove  $\text{Al}_2\text{O}_3$  template. The formation of the hybrid double wall nanotubes of conducting PPy or PT enveloped by magnetic Ni was investigated by using scanning electron microscope (SEM, JEOL JSM-5200). The structural properties of the hybrid double wall nanotubes were examined by using X-ray diffraction (XRD, Philips X'pert MPD X-ray diffractometer using a  $\text{Cu-K}\alpha$  ray) patterns and FT-IR (BOMEN MB 104) spectra. The magnetic properties of the systems were studied by using superconducting quantum interference device (SQUID, Quantum Design MPMS5) experiments.

## 3. RESULTS AND DISCUSSION

Figures 1 (a) and (b) show SEM photographs of the hybrid double wall nanotubes of PPy nanotubes enveloped by magnetic Ni nanotubes



**FIGURE 1** SEM images of the hybrid double wall nanotubes of PPy nanotubes enveloped by Ni nanotubes; (a) magnification of side view and (b) cross-sectional view.

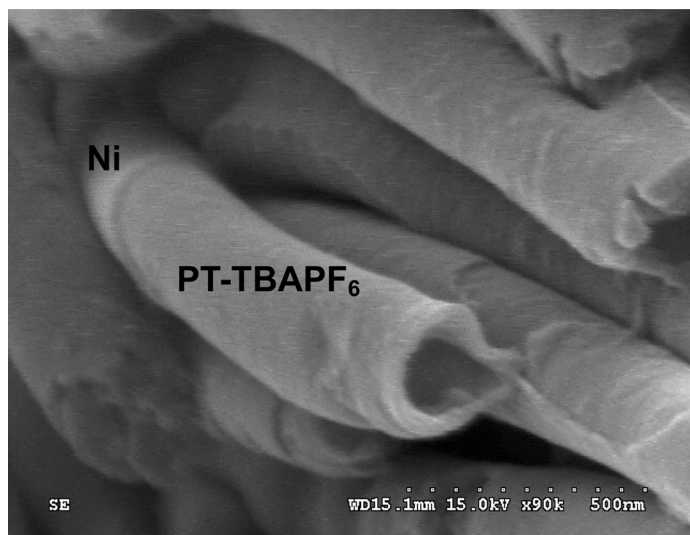
after removal of the  $\text{Al}_2\text{O}_3$  template using HF solvent. The formation of the hybrid double wall nanotubes of conducting polymer nanotubes enveloped by magnetic Ni nanotubes was confirmed by SEM images. We observed the open-ends of the hybrid double wall nanotubes as

shown in Figure 1(a). The hybrid double wall nanotubes was uniform and continuous, with length of  $\sim 40\ \mu\text{m}$ , as shown in Figure 1(b).

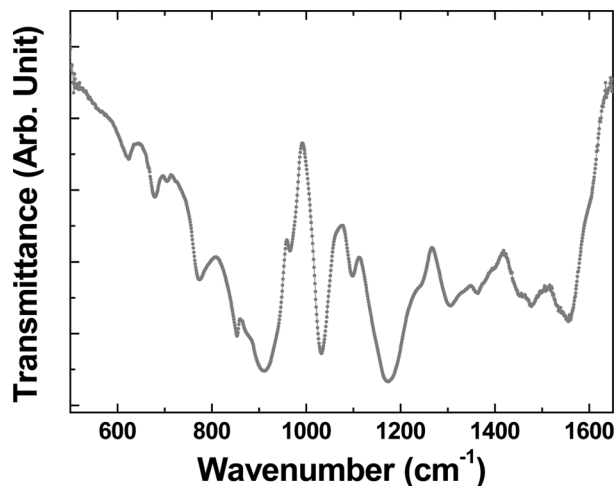
Figure 2 show SEM photograph of the hybrid double wall nanotubes of PT enveloped by magnetic Ni nanotubes after removal of the  $\text{Al}_2\text{O}_3$  template using HF solvent. Using the same synthetic condition of electrochemical deposition, we obtained the PT-Ni hybrid double wall nanotubes with open-ends and length of  $\sim 40\ \mu\text{m}$ .

Figure 3 show FT-IR spectrum of PPy nanotubes treated with HF solvent, which were synthesized at room temperature (RT). The band at  $1555\text{ cm}^{-1}$  and  $1474\text{ cm}^{-1}$  in the spectrum of PPy nanotubes corresponded to the C–C stretching vibrations and C–N stretching vibration in the pyrrole ring, respectively. The broad band between  $1418\text{ cm}^{-1}$  and  $1266\text{ cm}^{-1}$  was attributed to C–H or C–N in-plane deformation with a maximum at  $1303\text{ cm}^{-1}$ . The peak at  $1032\text{ cm}^{-1}$  was assigned to in-plane C–H stretching vibration. We observed the doping induced vibrations peaks at  $853\text{ cm}^{-1}$  and  $1098\text{ cm}^{-1}$ . The peak at  $620\text{ cm}^{-1}$  corresponded to the N–H out-of-plane vibration. From the FT-IR characteristic vibration peaks, we confirmed the polymerization of PPy nanotubes [5].

Figure 4 shows the XRD patterns of the hybrid double wall nanotubes of PPy and magnetic Ni nanotubes. Because conducting polymer has amorphous structure in general, the XRD peaks in Figure 4 originated

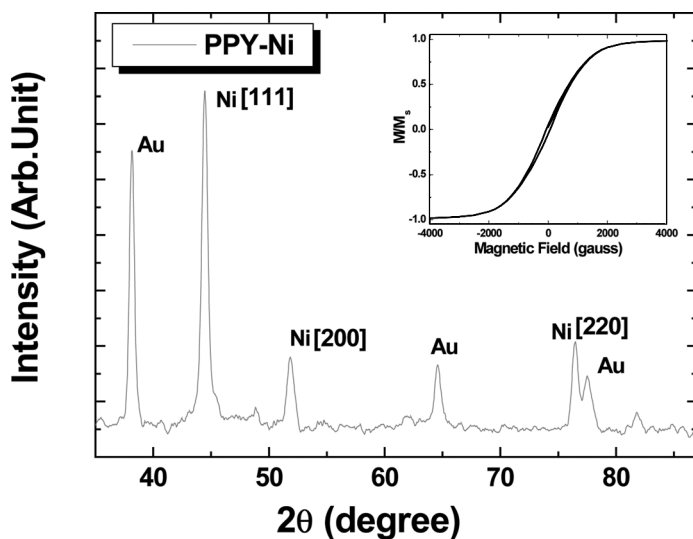


**FIGURE 2** SEM image of the hybrid double wall nanotubes of PT nanotubes enveloped by Ni nanotubes.



**FIGURE 3** FT-IR spectrum of PPy nanotubes.

from Ni or Au substrate, which were the typical face-centered cubic (FCC) structure. We observed the diffraction peaks at [111], [200], and [220]. The intensity of XRD peak at for [111] was much higher than that



**FIGURE 4** XRD pattern of the hybrid double wall nanotubes of PPy nanotubes enveloped by Ni nanotubes. Inset: Magnetic hysteresis curves of the hybrid double wall nanotubes of PPy and Ni.

of other peaks, indicating that magnetic Ni nanotubes have well grown in the direction of [111] [6]. The inset of Figure 4 shows the magnetic hysteresis curves of the hybrid double wall of PPy and magnetic Ni nanotubes, which were measured by using a SQUID at RT. From the hysteresis curves, we measured that the magnetic coercivity was 65 G [7], which implies that the PPy-Ni hybrid double wall nanotubes had ferromagnetic nature due to Ni nanotubes.

#### 4. CONCLUSION

We synthesized the hybrid double wall nanotubes of conducting polymer nanotubes enveloped by magnetic Ni nanotubes. Nanotubes of PPy and PT were synthesized in the nanoporous of  $\text{Al}_2\text{O}_3$  template through electrochemical polymerization method. Magnetic Ni nanotubes were fabricated by electrochemical deposition outside the wall of the conducting polymer nanotubes. From SEM images, we confirmed the formation of the hybrid double wall nanotubes. The length and diameter of the hybrid nano systems were  $\sim 40\text{ }\mu\text{m}$  and  $\sim 200\text{ nm}$ , respectively. From the XRD patterns, the Ni nanotubes in the hybrid nano-systems had well grown in the direction of [111]. The magnetic coercivity of the hybrid nano-systems was measured to be 65 G, implying ferromagnetic nature.

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