

Characterization and anticorrosion properties of carbon nanotubes directly synthesized on Ni foil using ethanol



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ABSTRACT

In this work, we describe the direct growth of carbon nanofilaments by the catalytic decomposition of ethanol on untreated polycrystalline Ni foil. Our work focuses on the effects of synthesis conditions on the growth of the carbon nanofilaments and their growth mechanism. Direct growth of carbon nanotubes (CNTs) is more favorable on lower-purity Ni foil. The highest yield was obtained at approximately 750 °C. The average diameter of the CNTs was approximately 20–30 nm. Raman spectra revealed that the increase of H₂ concentration in the carrier gas and synthesis temperature induced the growth of better-graphitized CNTs. Additionally, we investigated the anticorrosion properties of as-prepared products under simulated seawater conditions. The corrosion rate of the CNT/Ni foil system was maximally 50–60 times slower than that of the as-received Ni foil, indicating that the CNT coating may be a good candidate for corrosion inhibition.

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1. Introduction

Metal substrates have been widely used for practical applications because of their excellent electrical, thermal, and mechanical properties. However, their inherent weakness, susceptibility to attack by highly corrosive substances such as oxygen, acids, and salts, is still a challenge to overcome. [1]. Of various materials that have been suggested as protective coatings for metals [2–4], carbon nanostructures, such as carbon nanofilaments, diamond-like carbon, and graphene, are promising candidates for coating barriers against harsh, corrosive environments [5–7]. Coatings of carbon nanostructures are mainly achieved using spin coating, electrolytic deposition, and chemical vapor deposition (CVD) [8–10]. CVD is very useful for the direct synthesis of well-graphitized carbon nanostructures making it possible to achieve higher electrical conductivity and enhanced corrosion resistance. This method requires catalysts with high activity to obtain high-quality carbon structures

at low synthesis temperatures. Fortunately, metal substrates have some advantages for such direct formation. First, they can act as effective self-catalysts without additional catalytic agents. Second, adhesion between the substrate and the carbon nanostructures is adequate without the use of superfluous materials such as binders. Finally, the excellent thermal and electrical properties of metal substrates allow for enhanced performance of carbon nanostructure/metal hybrid systems.

Ni is one of the most commonly used metal substrates not only for the growth of carbon nanostructures, because of its excellent carbon solubility, but also for use as an electrode because of its superior electrical properties and low cost. Several groups have directly synthesized graphene on various types of macroscopic and polycrystalline Ni substrates, such as foil and foam, and demonstrated its effectiveness as a protective coating. Prasai et al. demonstrated enhanced corrosion resistance of graphene-coated Ni foil in aerated Na₂SO₄ solution [10]. Krishnamurthy et al. showed the superiority of graphene-coated Ni foam for prevention of microbially induced corrosion [11]. On the other hand, there is not much research applying carbon nanofilaments as anticorrosion coatings. Most studies have addressed the role of carbon nanofilaments as an effective additive to anticorrosion composite coatings [12,13]. To achieve thin and uniform coverage of carbon nanofilaments over Ni substrates, some researchers pretreated the surface

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of the substrates using mechanical polishing, oxidation, reduction, or chemical etching [14–17]. This is an effective way to maximize the amount of deposited carbon nanofilaments on Ni substrates, but the substrates can be seriously damaged during the pretreatment process, particularly at grain boundary regions, resulting in not only the deterioration of their original properties but also the acceleration of corrosion rate. Therefore, the search for direct and uniform coverage of Ni substrates with carbon nanofilaments without pretreatment processes is of importance for investigation of their feasibility as a protective barrier.

Herein, we report on the direct synthesis of carbon nanofilaments on untreated nickel foil using ethanol. First, we investigated whether substrate purity affects the direct growth. Also, we analyzed the effects of synthesis conditions, such as the H_2 concentration in the reactor, synthesis temperature, and synthesis time, on the growth of carbon nanofilaments. As-synthesized products were characterized in detail. It was found that the morphology and structure of carbon nanofilaments strongly depends on the synthesis temperature. In addition, we performed electrochemical analyses of the as-synthesized carbon nanofilaments/Ni-foil systems in simulated seawater to evaluate their corrosion resistance. As expected, the CNT coating layer remarkably reduced the corrosion rate of Ni foil. On the other hand, the presence of surface break-up, formed mainly at the boundary layer of Ni foil during synthesis, accelerated the corrosion rate.

2. Experimental

2.1. Materials preparation

Two types of nickel foil with different purities were used for the synthesis of carbon nanofilaments. Foil 1 had a purity of 99.98% (Aldrich), and Foil 2 had a purity of 99.2% (Surepure Chemetals, Inc.). The dimensions of the foils were 10 mm \times 20 mm \times 100 μ m. No mechanical or chemical pretreatment was performed on the substrates other than a simple cleaning. The substrates were first ultrasonically cleaned in an ethanol bath for 10 min in order to remove organic impurities from the surface; they were then dried

at room temperature. To condition metal substrates before growing CNTs, it is common practice to reduce substrates at temperatures ranging from 500 to 700 $^{\circ}$ C in H_2 for 1–3 h, but we did not carry out this pre-reduction process before the synthesis process.

2.2. Synthesis of carbon nanofilaments on Ni foil

The prepared nickel foils were placed on a quartz boat, and the boat was placed inside a quartz tube 32 mm in diameter and 400 mm in length with a wall thickness of 2 mm. The quartz tube was then installed in a chemical vapor deposition reactor and heated to the desired temperature at a rate of 10 $^{\circ}$ C/min under a carrier gas atmosphere. The synthesis temperature was controlled to be between 650 and 950 $^{\circ}$ C. Ar (flow rate = 100 cm^3/min) was used as the main carrier gas, and the concentration of H_2 in Ar was controlled to be between 0 and 90 vol%. Liquid ethanol (99.9%, Merck) was supplied as the carbon source. Ethanol is a good material because of its low cost, relative harmlessness, and ease of control. Ethanol was atomized by the ultrasonic system used in our previous work [18] and supplied at a flow rate of 5 cm^3/h into the reactor by the carrier gas. The synthesis time was controlled to be between 10 and 120 min. When the synthesis was completed, the ethanol and hydrogen flow was stopped sequentially. Then the reactor was cooled down to room temperature, while the flow of Ar was maintained at 100 cm^3/min . For comparison of corrosion properties, we also prepared a graphene-coated Ni foil (Gr/Ni) sample using a method suggested by a previous study [19].

2.3. Characterization

As-synthesized products were characterized using a variety of analytical techniques. The detailed components of the prepared Ni foils were confirmed by X-ray fluorescence analysis (XRF; Primus II, Rigaku Co., Japan). The measurement area was 30 mm^2 and the element line was $K\alpha$. X-ray diffractometry (XRD) was performed using a Rigaku DMAX-2500 system operated at 40 kV and 100 mA. Measurements were conducted over a 2θ range of 10–110 $^{\circ}$ with a $Cu K\alpha$ source emitting at approximately 1.54 Å . X-ray photoelectron spec-

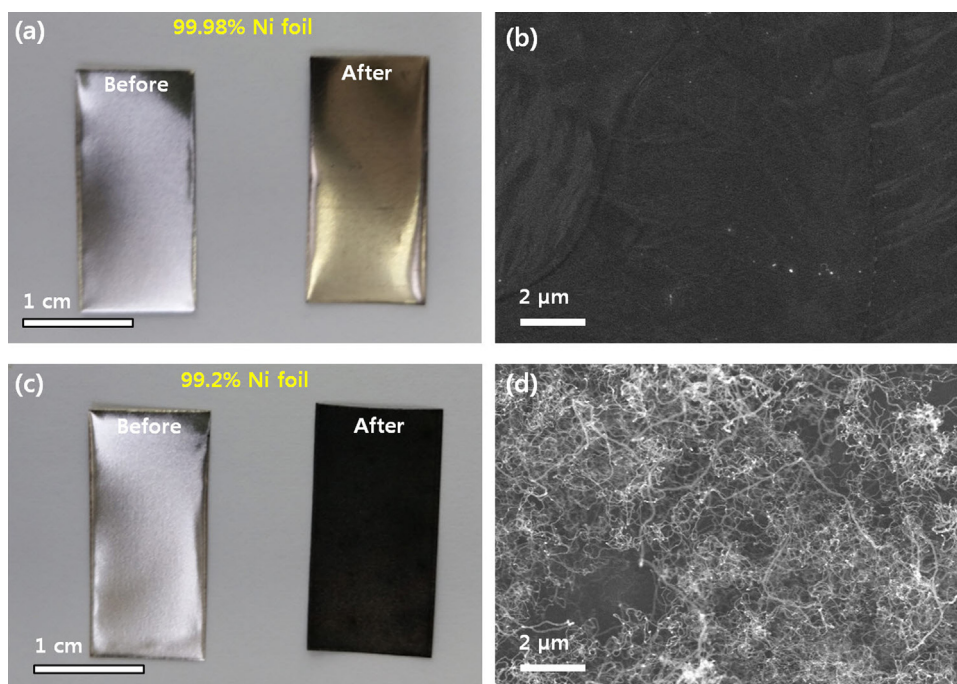


Fig. 1. Photos of (a) 99.98% Ni foil (Foil 1) and (c) 99.2% Ni foil (Foil 2) before and after synthesizing at 750 $^{\circ}$ C and for 60 min. (b, d) SEM images of (a) and (c), respectively.

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