JMEPEG (2012) 21:324–330
DOI: 10.1007/s11665-011-9958-4

1059-9495/$19.00

Preparation and Characterization of Nickel-Coated Carbon Fibers by Electroplating

Zhongsheng Hua, Yihan Liu, Guangchun Yao, Lei Wang, Jia Ma, and Lisi Liang

(Submitted May 2, 2010; in revised form April 13, 2011)

Electroplating technique was applied to coat carbon fibers with nickel. Before plating, the initial fibers were pretreated to improve the wettability in bath. The electroplating parameters were optimized to obtain high-quality nickel-coated carbon fibers, and the effects on plating were studied. The coated carbon fibers were characterized by SEM, XRD, and XPS. The coatings are uniform, smooth, bright, and adherent to carbon fibers not only along length but also along the diameter of the filaments, and mainly composed of pure nickel. Metal-carbon-oxygen bonds are present at the interface between nickel coatings and fibers, which provides the interfacial binding force. The results of performance tests showed that the nickel-coated fibers possess a good bonding strength not less than 78.5 kPa, and exhibit excellent oxidation resistance at high temperature. Compared with the initial fibers, the wettability with aluminum is also improved obviously.

Keywords advanced characterization, carbon fibers, coatings, electron microscopy, electroplating, nickel

1. Introduction

Carbon fibers (CFs) have been widely used as reinforcements in composite materials such as CFs reinforced metals, ceramics, plastics, and carbon-carbon composites due to the high specific strength, specific modulus, high thermal and electric conductivity, low expansion coefficient, and good self-lubricant (Ref 1-3). These properties are unaffected by temperatures up to more than 2000 °C in vacuum and inert atmospheres (Ref 4), but they are readily oxidized at temperatures as low as 400 °C in air. Meanwhile, as reinforcements, the poor wettability and harmful interfacial reaction between CFs and matrix material make it very difficult to obtain CFs reinforced materials with good interface bonding. Coating technology on CFs surface is an effective method to overcome the problems mentioned above.

A great deal of research on coating has been done. Generally speaking, coatings (Ref 5-14) on CFs include metal coatings, ceramic coatings, and composite coatings. Among these three coatings, the binding force between metal coatings and CFs is the strongest and the coatings are not easy to crack and abscise. The methods of metallization on CFs surface can be divided into physical methods including sputtering, iron plating, metal powders spraying, and chemical methods mainly including chemical plating and electroplating (Ref 15-20). Physical methods have strict requirements to apparatus, and the expense and energy consumption are higher. Thus, chemical methods are more applicable in practical operation.

The melting point of nickel is 1455 °C. After plating nickel, CFs can be well protected from oxidizing. At the same time, the wettability of CFs is also improved.

Compared with electroplating, electroless plating (Ref 21, 22) is more suitable for coating nickel alloys, such as Ni-P and Ni-W-P alloy, both of which have melting points lower than pure Ni. In this study, bright pure nickel was coated on CFs surface by electroplating. Particularly, it is a simple and inexpensive method to obtain uniform and firm coatings on CFs surface. The nickel-coated CFs can be widely applied to miniature capacitor of large capacitance, magnetic film, and many kinds of functional devices. Nickel-coated CFs and their applications have been studied widely (Ref 22-24), but, to our knowledge, aspects such as effect of process parameters on electroplating and analysis of the elements in coatings have rarely been focused on. In this article, emphasis is laid on these aspects.

2. Experimental

2.1 Pretreatment of CFs

The polyacrylonitrile (PAN)-based CFs used for coating in this article were provided by Liaoning Anke Activated Carbon Fiber Applyart Exploitation Inc. The fibers are long thread in series, with black and bright surface. It has 12k single filament per bundle, with the diameter about 7 μm. The properties of the CFs are shown in Table 1.

Thousands of single filaments cohere together and compose the CFs bundle by the adhesive force of organic glue. The glue membrane and some other grease infectants on the surface of CFs are both bad for electroplating. In order to obtain uniform coatings on individual fiber, CFs must be modified to improve their surface activity. In this study, the surface treatment of CFs included two steps: roasting in air at 400 °C for 20 min to

Zhongsheng Hua, School of Metallurgy and Resources, Anhui University of Technology, Maanshan 243002, China and School of Materials and Metallurgy, Northeastern University, 117 Box, Shenyang 110004, China; and **Yihan Liu, Guangchun Yao, Lei Wang, Jia Ma,** and **Lisi Liang**, School of Materials and Metallurgy, Northeastern University, 117 Box, Shenyang 110004, China. Contact e-mail: huazs83@yahoo.com.cn.

remove the organic binder on the surface of fibers, and then boiling in 40% (V/V) nitric acid solution for 40 min to activate the surface.

2.2 Preparation of Nickel-Coated CFs

Watts nickel plating solution was applied to coat CFs. Nickel sulfate ($NiSO_4 \cdot 6H_2O$, ≥99%) was used as the main nickel source. Nickel chloride ($NiCl_2 \cdot 6H_2O$, ≥98%) was used not only as a nickel source but also as anode activating agent. Boric acid (H_3BO_3, ≥99.5%) used as pH buffer. Dodecyl sodium sulfate ($C_{12}H_{25}NaS$, total alcohol ≥59%) was used as anti-pitting additive. Saccharin ($C_7H_5NO_3S$, ≥99%) and 2-butyne-1,4-diol ($C_4H_6O_2$, ≥98%) were used as the primary and the secondary brightener, respectively.

According to the designed bath formula, quantitative reagents were dissolved in distilled water, and then filtered. CFs acting as cathode were placed between two nickel anode plates in the bath (Fig. 1), and then direct current was imposed between the nickel anodes and the cathode. When electroplating process was finished, the nickel-coated CFs were washed in distilled water for several times, lastly dried in vacuum drying oven at 60 °C.

Table 1 The properties of CFs

Density, g/cm^3	Strength, GPa	Modulus, GPa	Specific resistance, $\Omega \cdot cm$	Carbon content, %
1.76	3.0 ± 0.2	230	10×10^{-3}	≥95%

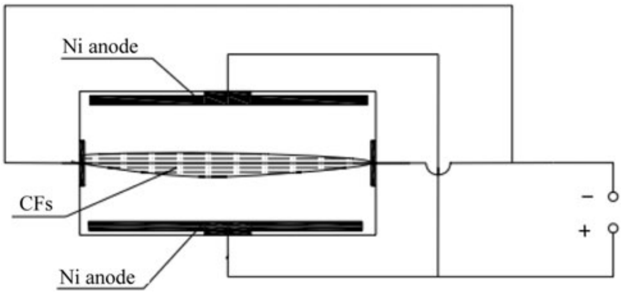

Fig. 1 The experiment device of electroplating nickel on CFs

2.3 Characterization and Test

SSX-550 scanning electron microscope (SEM) made in Japan was used for studying the morphology and thickness of the coated CFs. The phases of the coatings were characterized by PW3040/60 x-ray Diffractometer (Holand) using monochromatic target of Cu-Kα. XPS spectra were measured using ESCALAB250 spectrometer (USA) with Al-Kα = 1486.6 eV irradiation. The XPS spectra were taken from the surface of the coating to its interface with CFs. All spectra are represented in the binding energy scale that was obtained with respect to C1s peak of carbon at 284.6 eV. The software XPSPEAK4.1 was applied to analyze the detailed information about electron structure of coated fibers.

The oxidation behavior of uncoated and coated CFs was carried out in air at the heating rate of 5 °C/min using SDT Q600 thermoanalyzer (USA). The bonding strength of coated CFs was measured by thermal-cold cycling treatment. The wettability test was carried out using immersion method in molten aluminum.

3. Results and Discussions

3.1 Effect of Pretreatment on Electroplating

The initial CFs possess smooth surfaces, which cannot be separated well enough in the bath owing to hydrophobic interaction. The CFs are usually given a surface treatment to improve its wettability in the bath. Figure 2 shows the surface appearance of CFs before and after pretreatment. It can be seen that the smooth surface of CFs becomes rough, and the texture is deepened after pretreatment, accordingly, the specific surface area increases. Meanwhile, the results of XPS analysis (see Table 2) indicate that plenty of oxygen functional groups dominated by C-H and C-OH are present on the surface of CFs. These hydrophilic polar groups can effectively improve the wettability between CFs and bath, and act as the center of heterogeneous nucleation for nickel deposition. It was found that the unprocessed CFs are difficult to be plated with continuous and adherent nickel coatings, while the pretreated CFs can be completely wet in bath and the nickel coatings are continuous, and firmly adhered to CFs. The proper pretreatment contributes to obtaining the nickel-coated CFs with good bonding force.

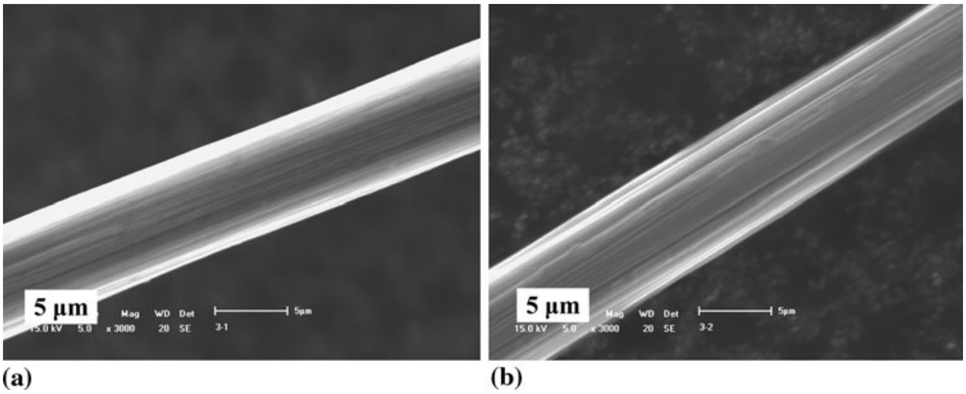

Fig. 2 SEM images of CFs before (a) and after (b) pretreatment

3.2 Effect of Process Parameters on Electroplating

As the nickel source, the concentration of nickel sulfate can vary within a large range from 100 to 350 g/L. At low concentration, the bath possesses good dispersancy, and the nickel grains in coatings are fine, but the deposition rate of nickel on CFs is relatively slow, and the cathode current efficiency is also low. It is quite the contrary while at high concentration. Nickel chloride plays an important role in activating anodes, besides being used as main salt and conductive additive. O_2 would generate if Cl^- in the bath is not adequate, and Ni^{2+} would be oxidized to form the insoluble Ni_2O_3, which deposits on nickel anodes preventing nickel from dissolving, and consequently the electroplating process is compelled to stop. It was found that the bath containing 250 g/L nickel sulfate and 40 g/L nickel chloride can work efficiently.

The acidity of bath is one of the most important factors to control the quality of nickel coatings. When $pH < 2$, cathode current efficiency is fairly low, only H_2 will be released, and nickel cannot deposit on CFs. When the pH value is extremely high, it is easy to produce $Ni(OH)_2$ colloid in the bath. The colloid can be taken into nickel coatings, which can make the coatings brittle consequently. Moreover, $Ni(OH)_2$ colloid can be adsorbed on nickel coatings to prevent H_2 from emitting out, and then leads to the increase of porosity in the coatings. In the bath, the concentration of Ni^{2+} is about 1.12 mol/L. The solubility product constant of $Ni(OH)_2$ $K_{sp}(Ni(OH)_2)$ is 5.5×10^{-16}. In order to prevent the formation of $Ni(OH)_2$, according to reaction (1) and formula (I), and the pH value of the bath should be lower than 6.35.

$$Ni(OH)_2 = Ni^{2+} + 2OH^- \quad \text{(Eq 1)}$$

$$K_{sp}\left(Ni(OH)_2\right) = \left(\text{Ref } Ni^{2+}\right)\left(\text{Ref } OH^-\right)^2 \quad \text{(Eq I)}$$

The pH value of the bath is rising gradually along with the process of the electrolytic reaction. It was found that the most appropriate pH value for electroplating nickel on CFs was 4-4.5. To maintain the pH value within the given range, H_3BO_3 was added into the bath. As buffering agent, H_3BO_3 have the most powerful buffering capacity when its concentration is more than 30 g/L.

The quality of nickel coatings was affected by the additives to a great extent. During the electroplating process, H^+ was reduced to form H_2 on the cathode, and the H_2 bubbles can be detained on CFs surface to form a shield, making nickel cannot deposit on CFs. Without adding additives, the CFs cannot be coated with intact nickel coatings. Seen from Fig. 3(a), serious plating leakage was present. After adding 0.1 g/L dodecyl sodium sulfate, the CFs were uniformly coated (see Fig. 3b). Dodecyl sodium sulfate in the bath can be absorbed on CFs, reducing the interfacial energy between CFs and bath, and thus H_2 bubbles can hardly

Table 2 Functional group analysis of carbon fibers after pretreatment with XPS

C-H		C-OH		C=O		COOH	
E_b	*a*, %	E_b	*a*, %	E_b	*a*, %	E_b	*a*, %
284.52	54.73	285.95	23.75	287.44	7.32	288.63	14.20

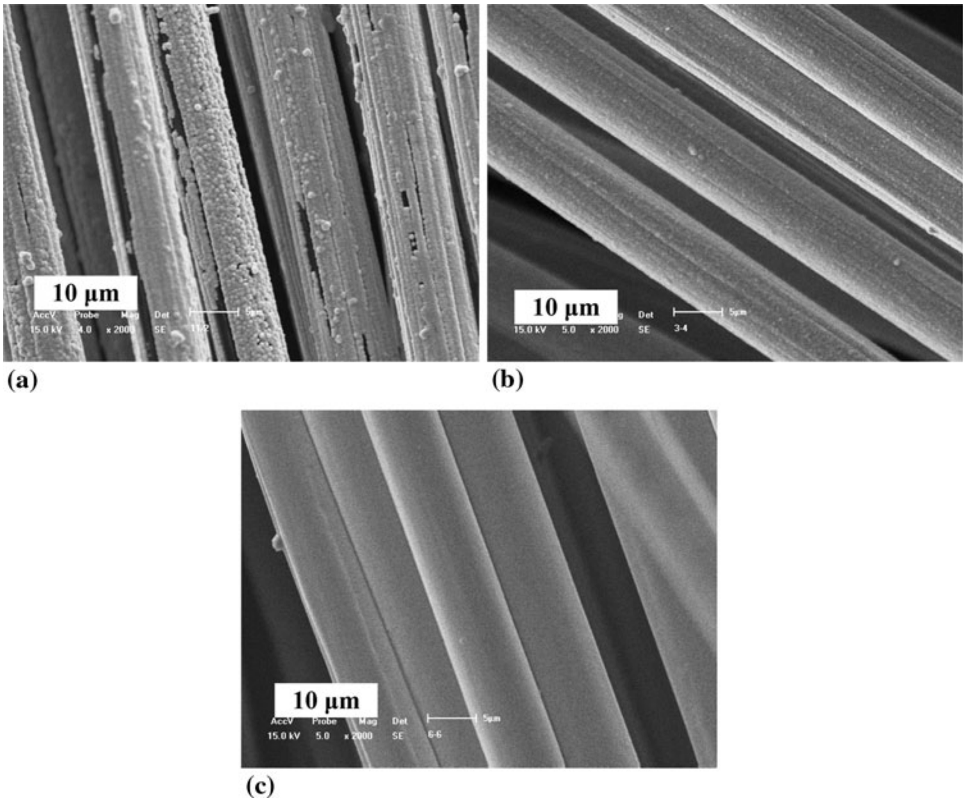

Fig. 3 SEM images of nickel-coated CFs: (a) no additives, (b) anti-pitting agent, and (c) anti-pitting agent and brighteners

absorbed on CFs, so pittings and plating leakages in coatings are avoided. In addition, after adding brighteners, bright, smooth, and grain-refining nickel coatings can be obtained (see Fig. 3c). Saccharin had an obvious effect on decreasing the grain size of nickel and making the coatings shiny, while 2-butyne-1,4-diol had a fairly brightening and leveling effect on coatings. Besides, by properly combining to use the two kinds of brightening agents, it can also improve the toughness and ductility of coatings.

Nickel electroplating can be carried out at 20-60 °C. Along with the rise of bath temperature, the solubility of nickel salts and the conductivity of electrolyte are both increased, which are favorable for achieving fast electroplating. At the same time, some detrimental effects also exist, for example, the saline hydrolysis and the precipitation reaction are aggravated, pittings in coatings increase, and the dispersibility of electrolyte decreases.

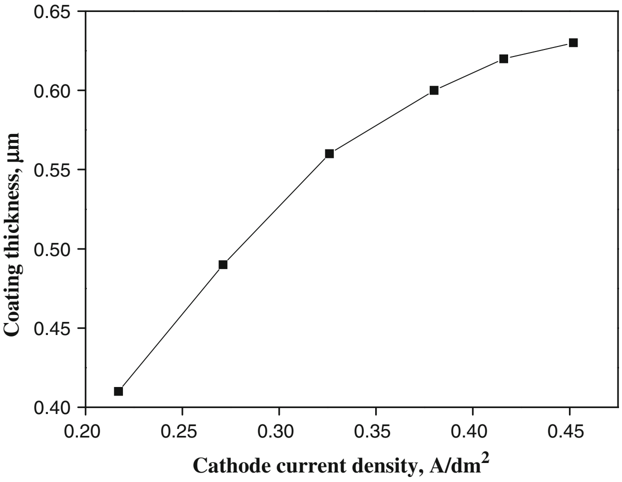

Fig. 4 Effect of cathode current density on coating thickness

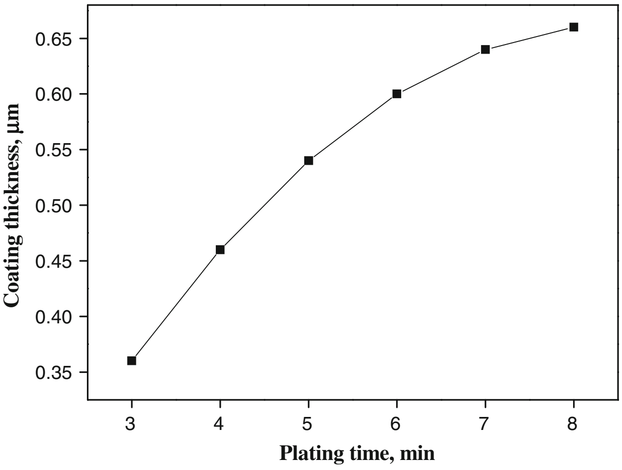

Fig. 5 Effect of plating time on coating thickness

The thickness of nickel coatings is mainly controlled by cathode current density and plating time. As shown in Fig. 4, by the current density increasing from 0.217 to 0.38 A/dm^2, the coating thickness increases fast; while the current density in excess of 0.38 A/dm^2, the curve become flat. It indicates that its effect on coating thickness is reducing by continuing improving the current density. When imposing high current, hydrogen evolution reaction on cathode is aggravated, and the current efficiency declines, resulting in decrease of thickening rate. What is more, owing to the larger resistance and worse heat dissipation performance of CFs, the nickel-coated fibers would be burnt and cohered together under high current density. Therefore, it is inadvisable to use high current density to plate nickel on CFs.

Figure 5 shows the effect of plating time on coating thickness. The coating thickness increases along with the increase of plating time, which can be explained by the step kinematics theory (Ref 25, 26), i.e., the nickel deposits on CFs are formed by layers, which is composed of many microscopic steps and the distribution of these microscopic steps on CFs is uneven. In growing process, these microsteps diffuse on the surface with different speeds, and the diffusion speed mainly depends on the step density. The higher the step density is, the less the atoms in unit step will be, which results in the reduction of diffusion speed. After nickel electroplating is carried out for several minutes, nickel deposits on CFs by granules, and tends to form dendritic crystals, which indicates that the steps density is increasing and reduces the growth speed of nickel. After a certain deposition period the Ni electrodeposition seems to stagnate and no obvious changes in the coating thickness could be observed. Moreover, plating time has significant influence on coating quality. It was found that the coatings were very thin with black core when the plating time was shorter. In contrast, the coatings became hard and brittle.

On the basis of a trial-and-error approach, the optimum process parameters of nickel plating are obtained as follows (see Table 3).

3.3 Characterization of Nickel-Coated CFs

3.3.1 SEM Investigations. The uncoated and coated CFs are, respectively, shown in Fig. 2(b) and 3. It can be seen that a great number of grooves which formed during the electroplating process are present on the surface of monofilaments along their lengths when no brighteners are added. The grooves disappear when two brighteners are used in plating. The cross section of nickel-coated CFs is shown in Fig. 6. Obviously, the thickness of the coating is uniform, and firmly adheres to CFs not only along length but also along the diameter of the filaments, and no bridging and no spalling of coatings are observed. The coating thickness is about 0.6 µm according to the SEM observation.

3.3.2 XRD Analysis. The results of XRD analysis clearly demonstrated the feasibility of electroplating nickel coatings on

Table 3 The optimum process conditions of nickel plating

$NiSO_4·6H_2O$	$NiCl_2·6H_2O$	H_3BO_3	$CH_3(CH_2)_1·SO_4Na$	$C_7H_5NO_3S$	$C_4H_6O_2$	pH	Temperature	Current density	Plating time
250 g/L	40 g/L	35 g/L	0.1 g/L	0.8 g/L	0.1 g/L	4-4.5	50°	0.38 A/dm^2	7 min

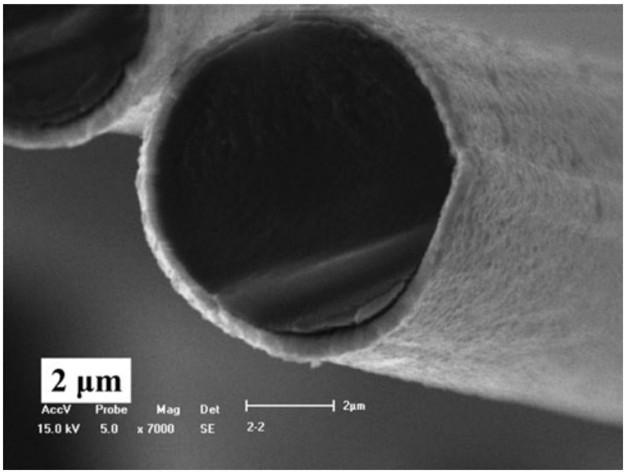

Fig. 6 SEM image of cross section of nickel-coated CFs

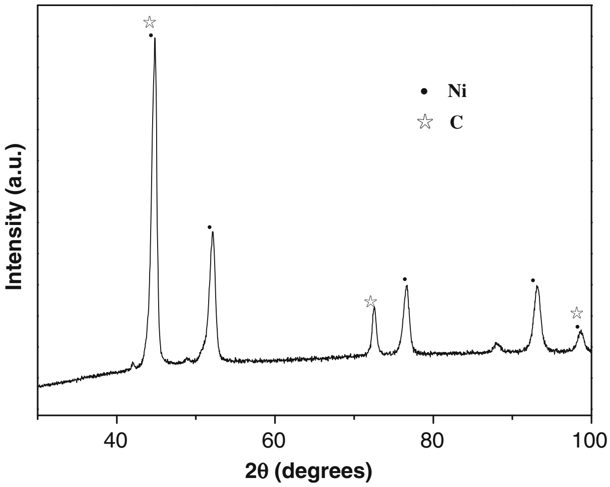

Fig. 7 XRD data of the nickel-coated CFs

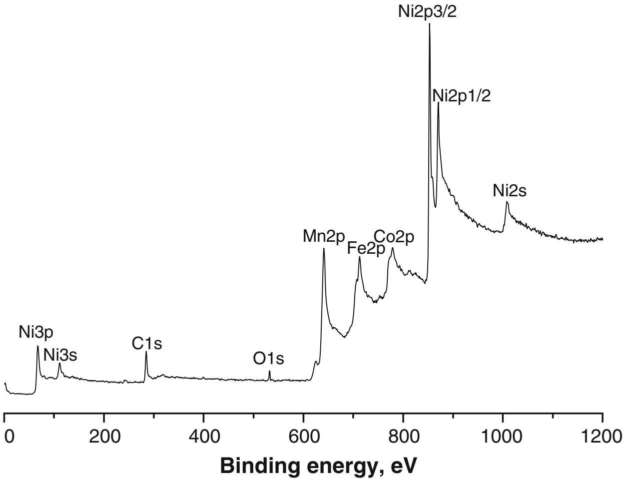

Fig. 8 Survey XPS spectrum of the nickel coatings on CFs

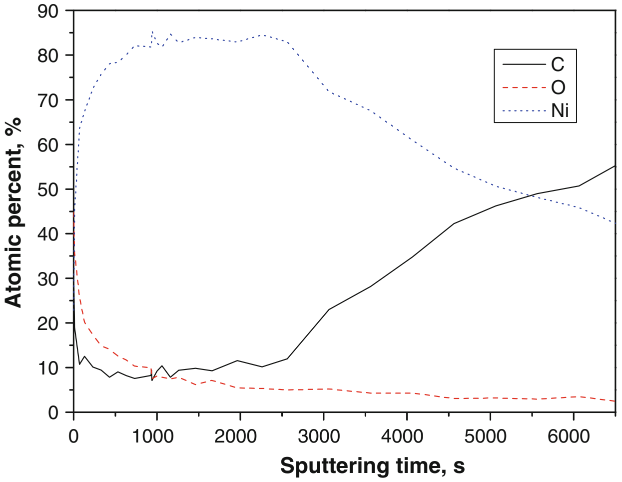

Fig. 9 Element percentages with sputtering time obtained by XPS

CFs. In comparison with the initial fiber, CFs were gradually becoming silver-gray and hard in plating process, indicating the formation of nickel on the fibers surface. According to the XRD patterns (Fig. 7), the composition of the coatings is represented mainly by Ni, and peaks belonging to other phases are not detected except C, which originates from CFs. It confirms that the coatings are fairly pure.

3.3.3 XPS Spectra of the Coatings. Survey XPS spectra of nickel-coated CFs are shown in Fig. 8. It reveals the presence of oxygen, carbon, and nickel, meanwhile, Mn, Fe, and Co which originate from the bath are present in the coatings as contamination. Figure 9 shows that on the coatings surface, the atomic ratio of O/Ni was higher than 1, for O_2 in the air can be absorbed on the surface of the coatings. With the increase of sputtering time, the content of Ni increases first and then decreases, while the change of C content is contrary to Ni, and the oxygen content is slowly decreasing to a very low level. That is in good accordance with the practical situation.

The C1s, O1s, and Ni2p spectral lines at the interface of nickel-coated CFs are shown in Fig. 10. The C1s spectrum gives evidence of mainly graphite-like structures as well as oxygen-containing carbon structures. The 532.1 eV component of O1s is a characteristic of oxygen-containing carbon structure. Those suggest that the carbon-oxygen bonds are present at the interface between CFs and nickel coatings. Deconvolution of the Ni2p spectrum results in detection of two doublets at 853.3 and 870.2 eV components for the coated CFs, which have the corresponding satellite line at 859.2 and 874.4 eV, respectively. The 853.3 eV component can be attributed to nickel in the metal-carbon bonds (Ref 27). Based on the XPS results, it can be concluded that metal-carbon-oxygen bonds are present at the interface, and it is these bonds that provide the binding force between nickel coatings and CFs. Simultaneously, the appearance of three features at 67.5, 111.7, and 1008.8 eV in the spectrum is observed. These reveal that the inner coatings are consisted of pure nickel. It is in excellent agreement with the results of XRD analysis.

3.4 Performance Test

3.4.1 Oxidation Test. The uncoated and nickel-coated fiber bundles were heated to 1300 °C in air in a thermoanalyzer. In comparison with the uncoated fibers, Fig. 11 shows the oxidation behavior of nickel-coated CFs. The uncoated CFs start to be oxidized at about 500 °C, and completely convert to CO and CO_2 at about 1000 °C, resulting in rapid mass

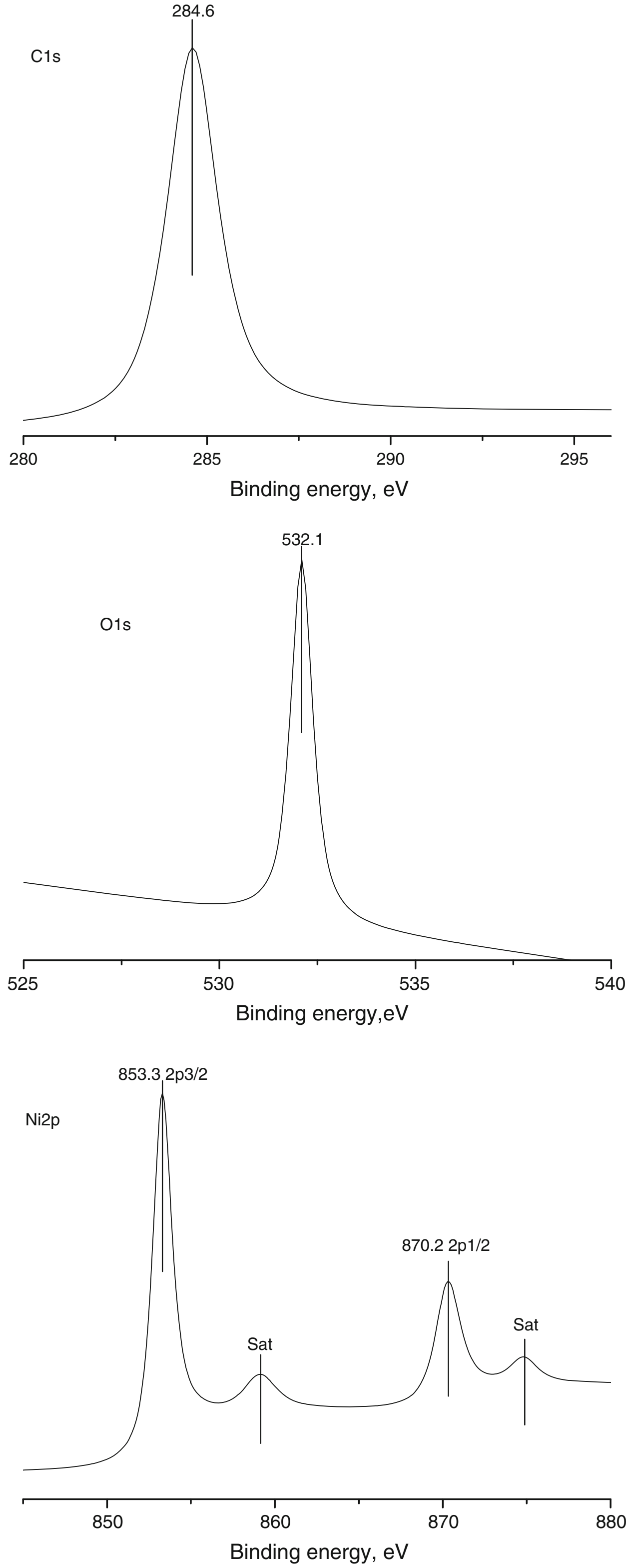

Fig. 10 The C1s, O1s, and Ni2p lines for coated CFs at the interface

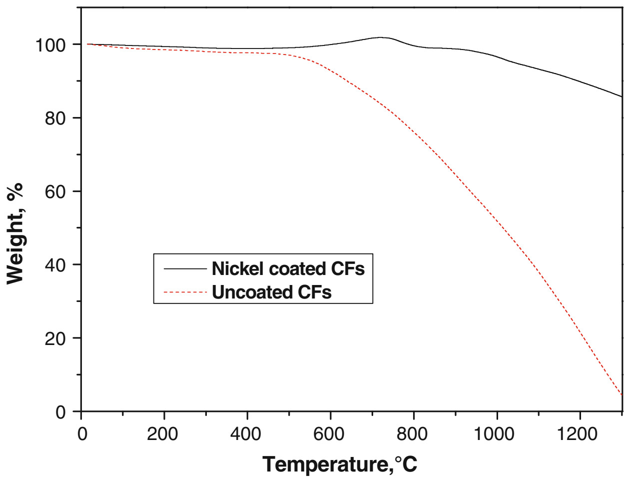

Fig. 11 Oxidation behavior of uncoated and nickel-coated CFs

decrease. As for nickel-coated CFs, along with the increase of temperature, the outer layer of the nickel coatings react with O_2 absorbed on their surfaces and begin to oxidize into NiO. As we know, the Pilling-Bedworth (PB) ratio of NiO is 1.52 (<2), and NiO film is a dense membrane. It is the NiO film that forms a protective layer on nickel to protect CFs against oxidation. In addition, owing to the absorption of O_2, the mass of nickel-coated CFs is increasing before 800 °C.

The radial thermal expansion coefficient of CFs between room and 900 °C is about $8\text{-}12 \times 10^{-6}\ ^\circ\mathrm{C}^{-1}$, while the axial thermal expansion coefficient is $-1.2\text{-}0 \times 10^{-6}\ ^\circ\mathrm{C}^{-1}$, which is much lower than that of nickel ($13 \times 10^{-6}\ ^\circ\mathrm{C}^{-1}$). A significant tensile stress will be built up between CFs and the coatings as the temperature changes owing to the thermal mismatch between the fibers and the nickel, and some coatings start to crack and abscise. The bare CFs are oxidized, and thus the weight begins to decrease. As shown in Fig. 11, only when the temperature is up to 1000 °C, the CFs begin to oxidize. At 1300 °C, the residual mass of nickel-coated CFs is still above 85%. It indicates that the oxidation resistance of CFs has been improved obviously after plating nickel coatings.

3.4.2 Bonding Strength of the Coatings. Owing to the specific characteristics, the bonding strength of nickel-coated CFs in this experiment was measured by hot-cold circulation method. The nickel-coated CFs were placed in the boiling water for 20 min first, and then immersed in ice water (0-5 °C) for 5 min. If no protuberances and no cracks are observed, the bonding strength would be not less than 78.5 kPa (Ref 28). The nickel-coated CFs were tested for six times with this method, and no cracks were observed under microscope. It confirms that nickel-coated CFs possess a good bonding strength, which is attributed to the metal-carbon-oxygen bonds formed at the interface between nickel and CFs.

3.4.3 Immersion Test. As reinforced material, it must possess good wettability. The wettability is normally measured by the sessile drop method, but it is not applicable to CFs for without leveling substrate. It is well known that CFs have bad wettability with aluminum. In this experiment, the wettability of uncoated CFs and nickel-coated CFs with aluminum melt were measured using immersion test.

The uncoated CFs and nickel-coated carbon fibers were immersed in molten aluminum for 10 s, respectively. When the uncoated CFs were introduced into molten aluminum, they were quickly rejected to the upside of the melt, nearly without adhering any aluminum, and their quality did not increase. While the nickel-coated CFs were immersed into the molten metal, a large amount of molten aluminum was adhered to the coatings, and the quality was increased about 30%, as described

Table 4 Weight change of CFs before and after dipping into molten aluminum

CFs type	Weight before dipping, g	Weight after dipping, g	Weight increasing rate, %
Uncoated	0.3819	0.3873	1.41
Nickel-coated	0.6571	0.8501	29.37

in Table 4. It is clear that the uncoated CFs do not hold any aluminum on their surface due to the bad wettability, but nickel-coated CFs adhered with a large quantity of aluminum, which indicates the best wetting property.

4. Conclusions

Based on the experimental results, the following conclusions can be drawn:

(1) Electroplating is an effective method to plate nickel on CFs. To obtain high-quality nickel-coated CFs, the influences on plating were discussed, and the optimum process conditions for plating nickel on CFs were obtained by trial-and-error approach. Under the optimum process conditions, uniform, smooth, and non-bridging bright nickel-coated carbon fibers were produced.

(2) After pretreatment, the wettability of CFs in bath has been improved. The nickel coatings are firmly adhered to CFs not only along length but also along the diameter of the filaments as confirmed with SEM observation. The coatings are mainly composed of pure nickel. XPS spectrum reveals that nickel-carbon-oxygen bonds are present at the interface between CFs and nickel coatings, which provide the interface binding force.

(3) The result of bonding strength test indicated that the bonding strength between the nickel coatings and CFs is not less than 78.5 kPa. Comparing with the uncoated fibers, the nickel-coated CFs possess better oxidation resistance at high temperature, and the wettability with aluminum is also improved obviously.

Acknowledgments

The study is financially supported by National Natural Science Foundation Key Project of China (No. 50834001) and National High Technology Research and Development Program 863 (No. 2009-AA03Z502). The authors are very grateful to Instrument Analysis Center of Northeastern University, for the help of analysis and test.

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