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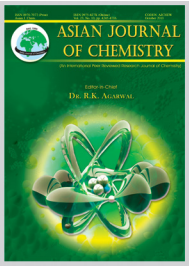

Effect of Ni Nanoprticles Coating on the Oxidation Behaviour of Carbon Fibers

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Carbon fibers with nickel nanoparticles coating were prepared by continuously electroplating. On the basis of thermal analysis of the samples with different nickel weight gain, it was found that the weight of nickel coating affected the oxidation behaviour of carbon fibers: when carbon fibers were coated with 2 wt % Ni, Ni nanoparticles catalyzed the oxidation of carbon fibers. On the contrary, when carbon fibers were coated with 25 wt % nickel, nickel coating protected carbon fibers, resulting in better oxidation resistance of carbon fibers.

Key Words: Carbon fibers, Electroplating, Nickel nanoparticles, Catalyzing, Oxidation resistance.

INTRODUCTION

Carbon fibers are widely used into resins and metals as reinforcements to fabricate high performance composites due to their excellent mechanical characteristics, such as high specific strength, specific modulus, low expansion coefficient and high thermal and electric conductivity[1-3]. However, a significant disadvantage of carbon fibers as reinforcing materials lies in their poor wettability as well as in their reactivity and strength loss when in contact with molten metals. Therefore, carbon fibers with metallic or ceramic coating as a protective barrier are prepared to resolve this problem[4]. With composite materials produced by certain processes and in operation in oxidizing media at elevated temperatures, this particular drawback markedly decreases the usefulness of carbon fibers[5]. Electrodeposition is a versatile and inexpensive technique to fabricate protective, conductive and decorative metallic coatings[6]. Electroplate nickel plating is a common protective coating of carbon fiber composite materials. Among the literature reports, most of the works are focused on the preparation process and few work pay attention to the oxidation behaviour of carbon fiber-coated Ni, especially of carbon fiber with low Ni weight gain coating.

In this work, we investigated the oxidation behaviour of carbon fiber-coated Ni. The distinct effect of Ni nanoparticles on the oxidation behaviour of carbon fibers which were coated by different Ni weight gain will be reported.

EXPERIMENTAL

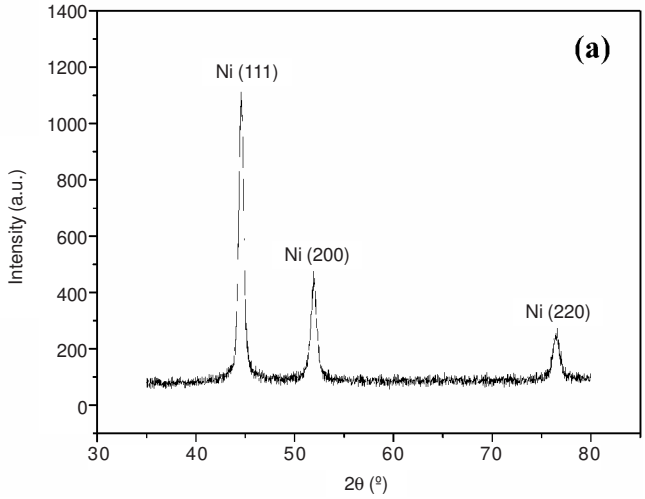
The carbon fibers used in the work were PAN-based carbon fibers, T300 and produced by the National Engineering Laboratory for Carbon Fiber Technology. The carbon fiber-coated Ni was prepared by continuously electroplating. The details of the electroplating bath were $NiSO_4 \cdot 7H_2O$ (270 g/L), $NiCl_2$ (70 g/L), H_3BO_3 (40 g/L) and $C_{12}H_{25}SO_4Na$ (0.1 g/L).

In the electroplating process, carbon fibers of different Ni weight gain were prepared by adjusting the electroplating time, respectively for 2 wt % and 25 wt %. After electroplating finished, the oxidation resistance of carbon fiber-coated Ni was studied under steady-state conditions in air respectively at 400, 500, 600, 700, 800 and 900 ºC for 10 min and the flow rate of air was 300 mL/min.

The evaluation of the mass loss was: after heated, the carbon fiber-coated Ni was acid-washed by HCl at 25 ºC. Because the physical and chemical properties of carbon fiber are stable in the HCl solution at 25 ºC, after acid-washed by HCl, the Ni particles on the surface of carbon fiber were dissolved and the weight of carbon fiber (without Ni coating) was scaled. The phase composition, surface morphology and microstructure of the coatings were studied by X-ray diffraction (XRD, Rigaku-D/max-λA) analysis and high-resolution scanning electron microscopy (SEM, JSM-6360LV), respectively.

RESULTS AND DISCUSSION

Fig. 1 shows the XRD patterns and SEM image of Ni coated carbon fibers before heating. Seen from Fig. 1(a), face centered cubic Ni presented in the prepared sample. The crystal grain size of Ni was about 14 nm estimated by Scherrer formula Fig. 1(b) showed the the carbon fiber was uniformly coated by Ni nanoparticles.



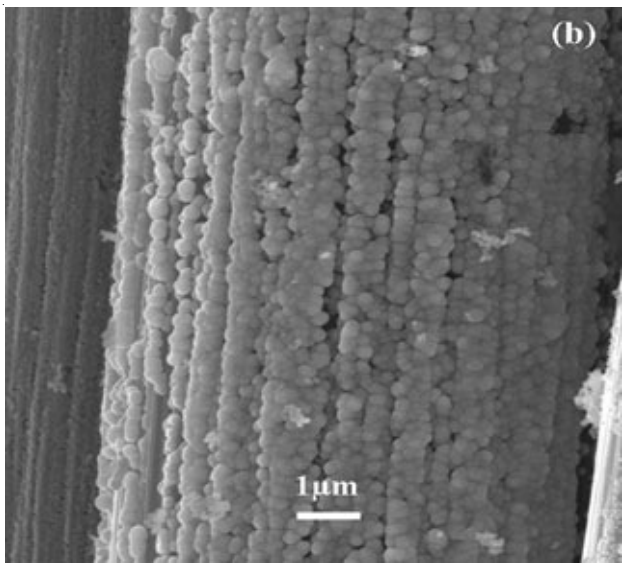


Fig. 1. Characterization of carbon fiber-coated Ni : (a) XRD patterns of carbon fiber-coated Ni and (b) SEM image of carbon fiber-coated Ni

Carbon fibers and Ni coated carbon fibers were heated in air respectively at 400, 500, 600, 700, 800 and 900 ºC for 10 min. Fig. 2 shows the mass loss of carbon fibers on different temperature. At 400 ºC, carbon fibers with 2 wt % gain had been oxidized resulting in the mass loss of 0.82 %, while 0.22 % of mass loss of carbon fibers without coating at 400 ºC and 0 % of mass loss of carbon fibers with 25 wt % gain at 500 ºC. Carbon fibers with 2 wt % gain were earlier and quicker to be oxidized than carbon fibers without coating. However, the oxidative stability of carbon fibers with 25 wt % gain was better than carbon fibers without coating.

On the basis of above thermal analysis, the weight of Ni coating affected the resistance of oxidation of carbon fibers. But at different Ni weight, the oxidation processes of carbon fibers were obviously not same with each other. This mainly depends on the distribution of Ni nanoparticles on the surface of carbon fibers. Nickel particles were distributed in different density on the surface of carbon fibers due to the different Ni weight gain of carbon fiber-coated Ni. The different distribution of Ni particles produced different effect on the oxidation behaviour of carbon fiber.

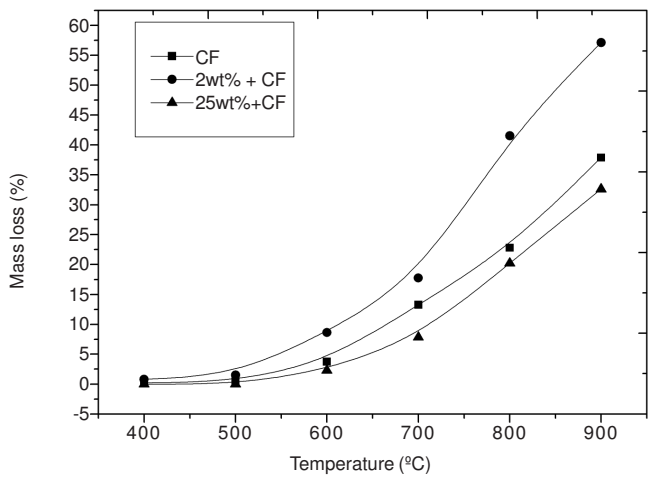


Fig. 2. Mass loss of the carbon fiber-coated Ni and carbon fibers

As the weight gain was 2 wt %, Ni particles were too sparse to cover the surface of carbon fibers, as observed from Fig. 3 (a). For such situation, carbon fibers could directly contact with oxygen. It is well known that Ni particles between 10 and 100 nm are very effective in catalyzing the graphitization of carbon. In reality carbon fibers were catalytically oxidized by the Ni nanoparticles *via* (1) diffusion of carbon into Ni, eventually forming Ni-carbide and (2) decomposition reaction of Ni-carbide with oxygen in air into Ni particles, CO, CO_2 and c-layer[7-9], as observed from Fig. 3(b). Since Ni particles were distributed too sparsely to gather and agglomerate, the Ni nanoparticles catalyzed the oxidation of carbon fibers continuously during the oxidation process. The catalytic process was more active as the temperature rising. Therefore, carbon fibers with 2 wt % gain were earlier and quicker to be oxidized than carbon fibers without coating from 400 to 900 ºC.

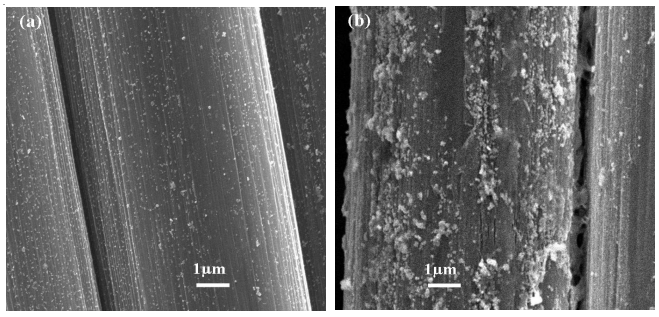


Fig. 3. SEM images of carbon fibers with 2 wt % gain: (a) before heated and (b) heated by 800 ºC

As the weight gain was 25 wt %, Ni particles seemed to joint closely indicating good attachment to cover the carbon fibers perfectly [Fig. 4(a)]. The Ni coating can protect carbon fibers from oxygen present in the air. At the beginning of oxidation, the out layer of the Ni particles coating was oxidized to nickel oxide film [Fig. 4(b)]. During this period, carbon fibers were protected from oxygen by the Ni coating and nickel oxide film, so the oxidation resistance of carbon fibers with 25 wt % gain was better than carbon fibers without coating. With the rise of the temperature, the greater depth of penetration of carbon into Ni resulted in the weaker bonding of carbon fiber and Ni coating; the density of Ni coating was

poor because Ni particles gathered and grew[10,11] [Fig. 4(c)]. Finally, carbon fibers began to be oxidized due to oxygen penetrating into Ni coating and reacting with carbon fiber; then Ni coating cracked and exfoliated [Fig. 4(d)]. Therefore, the beginning temperature to oxidation of carbon fibers with 25 wt % gain was higher than that of carbon fibers without coating because of the protection of the Ni coating; as Ni particles gathered and grew, the oxidation process of carbon fibers with 25 wt % gain was similar to that of carbon fibers without coating because Ni particles were too large to catalyze the oxidation of carbon fibers.

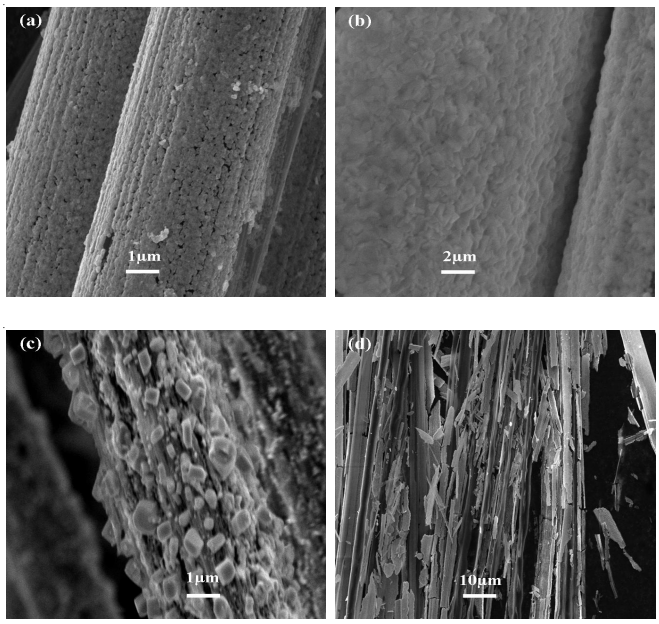


Fig. 4. SEM images of carbon fibers with 25 wt % gain : (a) before heated; (b) heated by 600 ℃; (c) heated by 800 ℃ and (d) heat by 900 ℃

Conclusion

In conclusion, Ni coating played different roles in the oxidation of carbon fibers as the different distribution of Ni particles on the surface of carbon fibers. Nickel particles sparse on the surface can catalyzed the oxidation of carbon fiber, resulting in earlier and quicker oxidation like carbon fibers with 2 wt % gain. Nickel particles covered the surface can protect the carbon fiber, resulting in better resistance of oxidation like carbon fibers with 25 wt % gain. In particular, carbon fibers with 2 wt % gain of distinct tendency to oxidize should provide a new practical direction of carbon fiber-coated nickel.

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