Composites Part B 76 (2015) 286–291

Contents lists available at ScienceDirect

Composites Part B

journal homepage: www.elsevier.com/locate/compositesb

Influence of electroless nickel-plating on fracture toughness of pitch-based carbon fibre reinforced composites

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ARTICLE INFO

Article history:
Received 12 September 2014
Received in revised form
26 November 2014
Accepted 31 January 2015
Available online 10 February 2015

Keywords:
A. Carbon fibre
A. Polymer–matrix composites (PMCs)
B. Fracture toughness
E. Surface treatments

ABSTRACT

Nickel-Pitch-based carbon fibres (Ni-PFs) were prepared by electroless nickel-plating to enhance fracture toughness of Ni-PFs reinforced epoxy matrix composites (Ni-PFs/epoxy). The surface properties of Ni-PFs were determined by scanning electron microscopy (SEM), X-ray photoelectron spectrometry (XPS), and X-ray diffraction (XRD). The fracture toughness of the Ni-PFs/epoxy was assessed by critical stress intensity factor (K_{IC}) and critical strain energy release rate (G_{IC}). The fracture toughness of Ni-PFs/epoxy was enhanced compared to those of PFs/epoxy. These results were attributed to the increase of the degree of adhesion at interfaces between Ni-PFs and matrix resins in the composites.

1. Introduction

Carbon fibres have been generally used in high performance composite materials because of their outstanding properties such as superior mechanical properties, high temperature resistance, chemical stability, and light weight. These excellent properties of carbon fibres are transferred to matrix through their interface. Accordingly most of carbon fibres reinforced composite properties are controlled by interfacial adhesion between carbon fibres and matrix resins [1–4]. In order to improve interfacial adhesive strength between carbon fibres and matrix resins, the carbon fibre surface is commonly modified by numerous surface treatments such as plasma [5], grafting [6] and metal plating [7]. These treatments tend to increase active sites and wettability of the carbon fibre surface and consequentially enhance the mechanical interfacial properties of the composites [8–13].

Of the effective surface treatments, Metal plating is a method of making a metal surface covering on the carbon fibres and has been used to improve fracture toughness of carbon fibre reinforced composites. Metal plating methods may be classified into electroplating and electroless plating. Electroplating is performed when metal ions are reduced to the metallic state and deposited as such at the cathode by using electrical energy [14]. Electroless plating is performed when exposed to plating solutions coating molten salt in the presence of a reductant. In contrast electroplating, it is not necessary to send an electric current through the solution to form a deposit [15,16].

Although copper, gold and silver can also be used as main materials in electroless plating, the most common electroless plating method is electroless nickel plating because of its low cost. The nickel plating process is based on a redox reaction in which the reducing agent is oxidized and Ni_2C ions are reduced on the substrate surface. Once the first layer of nickel is deposited on base materials it acts as a catalyst for the process. As a result, a relationship between plating thickness and time usually occurs. *Abraham* et al. [17] reported that the plating thickness is nanoscale. If the reducing agent is sodium hypophosphite, the obtained deposit will be a nickel–phosphorus (Ni–P) alloy. The as-deposited Ni–P alloys were reported to have a nonequilibrium phase structure. It is generally accepted that a microcrystalline, amorphous, or a coexistence of these two phases can be obtained depending on phosphorus content [18–20].

The objective of this study is to evaluate the influence of the electroless nickel-plating on the pitch-based carbon fibre surface properties and on the fracture toughness of nickel-plated pitch-based carbon fibre reinforced epoxy matrix composites. The fibre surfaces were characterized by SEM, XPS and XRD to confirm the morphology of the fibre surfaces and the elemental compositional

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http://dx.doi.org/10.1016/j.compositesb.2015.01.052
1359-8368/

changes. The critical stress intensity factor (K_{IC}) and critical strain energy release rate (G_{IC}) were investigated to determine the fracture toughness of the pitch-based carbon fibre reinforced composites.

2. Experimental

2.1. Specimen preparation

The epoxy resin used in this work was diglycidyl ether of bisphenol A (DGEBA, YD-128) which was supplied by the Kukdo Chem. Co., Korea. The curing agent used in this work was a modified cycloaliphatic amine curing agent (KH-819) supplied by the Kukdo Chem. Co., Korea. Pitch-based carbon fibres (PFs) were supplied by GS Caltex Co., Korea.

The schematic diagram of the electroless nickel-plating processes involved in the activation and metal deposition is illustrated in Fig. 1(a). Before the electroless nickel-plating process, the PFs were pretreated in 5 M HNO_3 for 30 min in order to increase interfacial adhesion between the nickel and the PFs. They were sequentially activated in tin chloride ($SnCl_2$) and palladium chloride ($PdCl_2$) solution for 30 min each. By activating, Sn/Pd nucleates adhered to the surface of PFs, and these nucleates accelerated the metal plating during the electroless nickel-plating. Ni-PFs were obtained by dipping the PFs into the nickel bath for 60–600 s. The constituents of plating solutions and reaction conditions are listed in Table 1.

The diameter and length of Ni-PFs obtained after plating were 30–60 μm and 400–700 μm respectively. The 5 wt% of Ni-PFs was dispersed within the epoxy resin for 1 h using a 3-roll-mill at room temperature. The mixtures were then poured into a mould and cured at 100 °C for 1 h and 130 °C for 1 h. The preparation process of Ni-PFs/epoxy composites and photographs of dispersion state of Ni-PFs before and after using 3-roll mill are presented in Fig. 2.

2.2. Characterization

The morphologies of the Ni–PFs were measured by a scanning electron microscope (SEM, JSM-6701F, JEOL). The surface properties of the Ni–PFs were characterized by X-ray photoelectron spectroscopy (XPS, K-alpha, Thermo Scientific). The XPS experiment was performed using a K_α spectrometer equipped with an AlK_α X-ray source. The base pressure in the sample chamber was controlled in the range from 10^{-8} to 10^{-9} torr. The X-ray diffraction (XRD) patterns of these samples were obtained using a Bruker

Table 1
Composition and conditions of electroless Ni plating bath.

Composition & condition	
$NiSO_4 \cdot 6H_2O$	280 g/L
$NiCl_2 \cdot 6H_2O$	40 g/L
$Na_3C_6H_5O_7 \cdot 1.5H_2O$	15 g/L
$NaH_2PO_2 \cdot 2H_2O$	100 g/L
NH_4Cl	100 g/L
$PbNO_3$	30 g/L
pH	8.25
Temperature (°C)	90 ± 1
Plating time (sec)	0–600

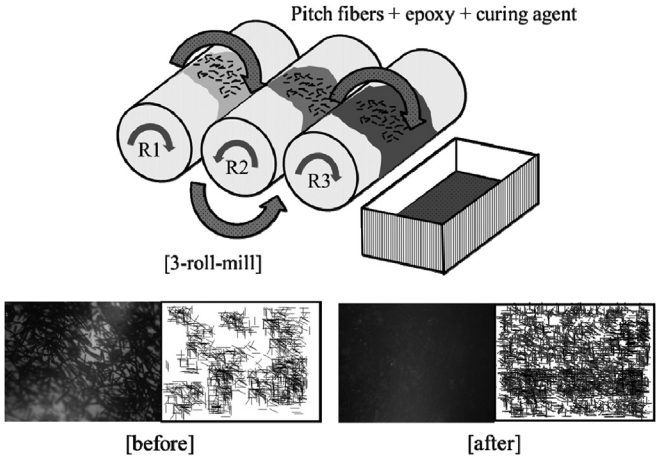

Fig. 2. The preparation process of Ni-PFs/epoxy and pictures of dispersion state of Ni-PFs in composites before and after using 3-roll-mill.

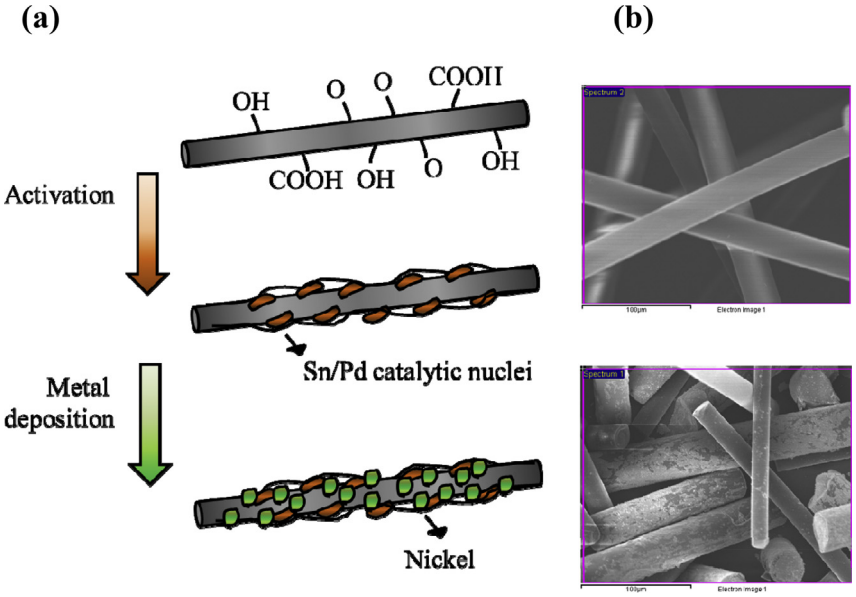

Fig. 1. (a) Schematic diagrams of the electroless Ni-plating processes involved in the activation and metal deposition; (b) SEM photographs of as-received PFs and 300 Ni-PFs.