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Enhanced mechanical strength of carbon fiber/magnesium alloy composites using nano-metal plating and surface fluorination

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ABSTRACT

Carbon fibers could be deposited uniformly with duplex coatings of nickel (the internal layer) and cupper (the external layer) using an electroplating process. The thickness of Ni and Cu was about 1-2 ㎛, respectively. The surface modification of Cu-Ni coated carbon fibers depended on the fluorination temperature; the CuO layer on Cu film was decreased by surface fluorination at 25℃, and the CuF_2 was prepared by surface fluorination at 100℃. The surface fluorination caused to the increasing of surface roughness. Consequently, the adhesion between fluorinated Ni-Cu-Carbon fibers and Mg alloys could be improved, and it led to the improvement of tensile strength of carbon fibers reinforced magnesium matrix composites.

1. INTRODUCTION

Carbon fiber reinforced metal (CFRM) matrix composites have been extensively used in a wide range of industrial applications because of their outstanding mechanical and/or physical properties [1, 2]. Among them, carbon fiber reinforced magnesium (C/Mg) composites have been structural element applications in high-precision space based systems [3]. Comparing with existing materials, such as carbon fiber reinforced epoxy matrix composites, C/Mg can offer significant gains in terms of specific strength and modulus, combined with the inherent environmental stability of metallic system. Magnesium has higher thermal and electrical conductivity than conventional organic materials, which allied to its high temperature capability results in composites with superior properties which are insensitive to temperature over the medium temperature range. However, weak bonding between carbon fiber and base metal or alloy makes these composites to poor mechanical properties. Especially, in the Al–C fiber composites, the existence of the reaction products such as Al_4C_3 at the interface is a drawback associated with metal matrix composites (MMCs) synthesized via liquid state processing [4]. It has been widely reported that the formation of this brittle compound at the interface has an adverse affect on the mechanical properties of the MMCs [5]. Also the carbon fibers are not wetted and not dispersed well in the melted aluminum [6]. To improve the wetting and dispersion between carbon fibers and base metal/alloy, two

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[1), 2)] Graduate Student, [3),4),5)] Professor techniques regarding metal nano-plating [7] and powder metallurgy [8] with hot pressing were applied to prepare CFRM matrix composites in this study. Accordingly, the primary aim of the present study is to synthesize magnesium alloy composite materials reinforced with carbon fibers having high mechanical strength. Especailly, in this study, the effect of surface fluorination on the adhesion between metal plated carbon fibers and base Mg alloy.

2. EXPERIMENTAL DETAILS

In this study, magnesium alloy AZ91D (supplied by Sakai Ovex Co., Ltd., 100–200 ㎛) powders and Ni–Cu coated carbon fibers (provided by TohoTenaxCo.,Ltd., 7 ㎛$^{\phi}$ x 50mm^{l}) were used as the matrix material and the reinforcing fibers, respectively. AZ91D alloy has high modulus and tensile strength and has been used as the matrix material in synthesizing MMCs. The Ni and Cu coatings were deposited on carbon fibers using the electroplating process. After surface acid treatment with hydrochloric acid (0.1M), the carbon fibers were designed to successively pass through two plating baths of Ni and Cu. Namely, the carbon fibers can be coated with duplex coatings of nickel (the internal layer) and copper (the external layer), that is denoted by Cu–Ni–C. The Ni and Cu plating bath was prepared using 20g/dm^3 nickel(II) sulfate hexahydrate (Nacalai Tesque Inc.), 20g/dm^3 cupper(II) sulfate pentahydrate (Nacalai Tesque Inc.), 30g/dm^3 tri-sodium citrate dehydrate (Nacalai Tesque Inc.) and sodium ammonium soulution (Kanto Chemical Co. Inc.) as a pH adjuster. Then sodium phosphinate monohydrate (Nacalai Tesque Inc.) was used as a reducing agent. The electroplating process was controlled to 55℃ and pH 5.0 with 0.5 A/dm^2 of current density. Finally, the substrate was rinsed carefully with ion-exchanged water and dried in a 70℃ air chamber after filtering. After Cu-Ni-Carbon fibers were acquired, they were blended 2 wt% with Mg alloy particles in ball milling machine for 2h. The blended powder mixture (2g) was compacted to a billet (20×60×1 mm) at a pressure of 2.5 MPa and 673 K for 1h under 10%H_2–90%N_2. Namely, the magnesium matrix composites were synthesized under hot-pressing using powder metallurgy method. Regarding surface fluorination, details of the fluorination apparatus and process have been given in our previous paper [9, 10]. Fluorinated Cu-Ni-C fibers were prepared by direct fluorination using F2 gas under various reaction conditions. Reaction temperature, fluorine pressure, and reaction time were set at 25-100℃, 101 kPa, and 1 h, respectively. Surface and cross-section morphology of Cu–Ni–Carbon fibers reinforced magnesium matrix composites were observed using SEM–EPMA (S-2400; Hitachi Ltd.). The tensile strength of composite fibers was investigated using a MTS 4482 (Instron Corp.).

3. RESULTS and DISCUSSION

Fig.1 shows SEM image (a) and mapping images ((b)Ni, (c)Cu and (d)C) of Ni-Cu composite film coated on carbon fiber in cross-section of magnesium composite fiber. It was found that the Ni and Cu films of 1.5 ㎛ thickness, respectively, covered uniformly on the carbon fibers as shown in Ni and Cu mapping images. Comparing with casting

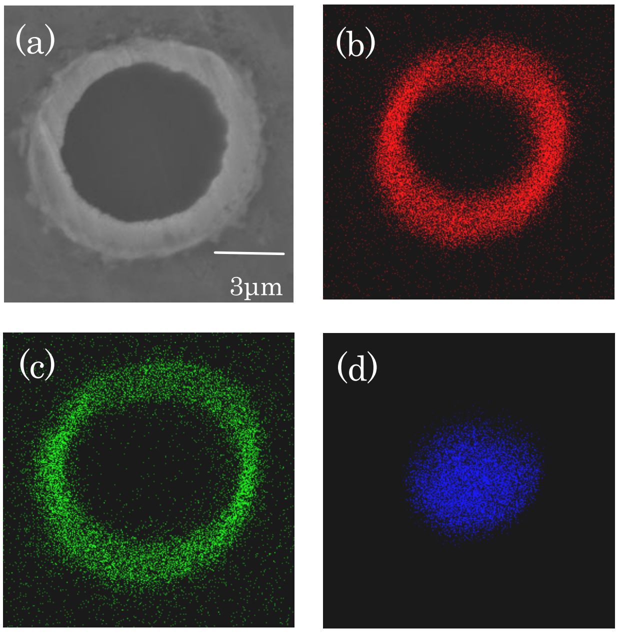

Fig.1 SEM image (a) and mapping images ((b)Ni, (c)Cu and (d)C) of Cu–Ni duplex layers coated on carbon fiber in cross-section of magnesium composite matrix.

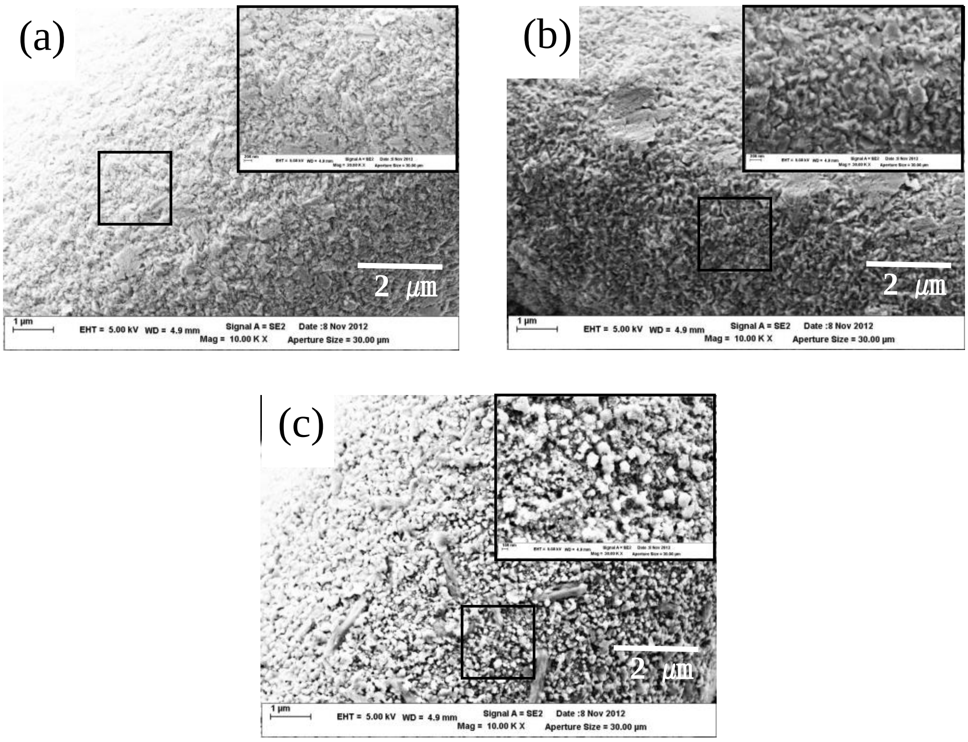

Fig.2 SEM images of Cu-Ni-C fibers prepared with (a) untreated, (b) fluorinated at 25℃, and (c) fluorinated at 100℃.

preparation method, the coated metal films existed stably on carbon fibers without any diffusion to outer base Mg alloy.

Fig.2 shows SEM images of Cu-Ni-C fibers prepared with (a) untreated, (b) fluorinated at 25℃, and (c) fluorinated at 100℃. With increasing the fluorination temperature, the surface roughness increased. Namely the surface fluorination could affect the surface morphology of Cu-Ni-C fibers.

Fig.3 indicates XPS spectra of Cu 2p for various samples prepared with (a) untreated, (b) fluorinated at 25℃, and (c) fluorinated at 100℃. All binding energies were calibrated to the C 1s peak at 284.8 eV of carbon. Cu and CuO peaks located at the binding energy (BE) of 933 eV and 934.8 eV were detected respectively in untreated sample (a). After fluorination at 25℃, the CuO peak decreased as shown in Fig. 3 (b). Furthermore, when the fluorination temperature increased to 100℃, the peak of CuF_2 was newly created whereas the peaks of Cu and CuO decreased. In the SEM images of fluorinated samples, the increasing of surface roughness may be reasoned for the conversion of CeF_2 from CuO existed on Cu metal.

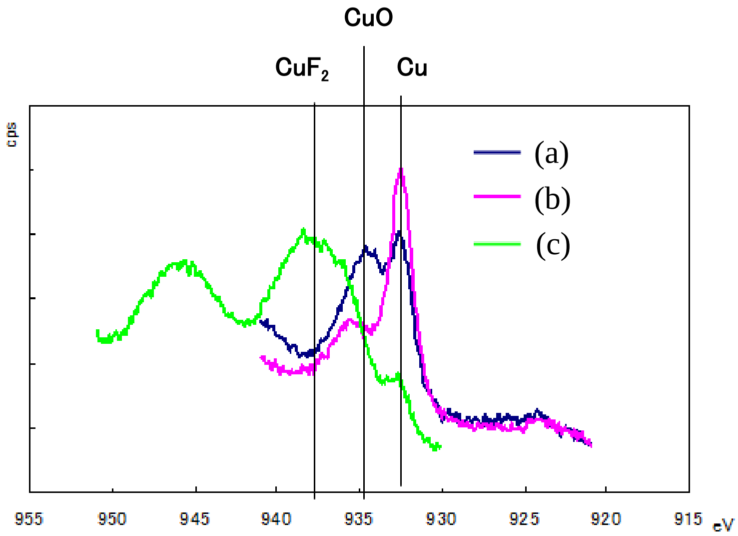

Fig.3 XPS spectra of Cu 2p for various samples prepared with (a) untreated, (b) fluorinated at 25℃, and (c) fluorinated at 100℃.

The stress-strain curves from tensile testing of (a) only Mg alloy, (b) untreated carbon fiber reinforced magnesium alloy composites, (c) Cu-Ni-C magnesium alloy composites, (d) composites of (c) fluorinated at 100℃ are shown in Fig.4. Ultimate tensile strength (UTS) values of sample (b) was 107 MPa even lower than that (136 MPa) of sample (a). It may be reasoned for the low affinity between carbon fibers and base Mg alloy. However, the UTS value (157 MPa) of the duplex Ni-Cu coated carbon composites (c) increased comparing with those of sample (a) and (b). Namely, metal coating between carbon fibers and base Mg alloys led to the improvement of tensile strength of composites. Furthermore, the UTS value of Cu-Ni-C and Mg composites was increased to 190 MPa by the surface fluorination, as shown in Fig.4 (d). It may be reasoned for the elimination of oxide films on Cu and the increasing of surface area of Cu layer. Namely, the adhesion between Ni-Cu-C fibers and Mg alloys could be improved by these effects of surface fluorination.

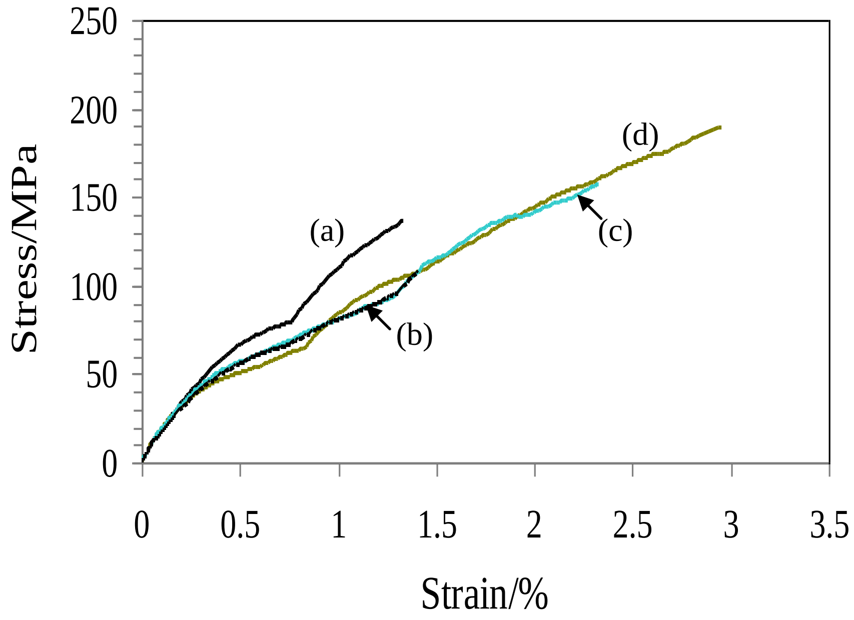

Fig.4 Stress-strain curves from tensile testing of (a) only Mg alloy, (b) untreated carbon fiber reinforced magnesium alloy composites, (c) Cu-Ni-C magnesium alloy composites, (d) composites of (c) fluorinated at 100℃.

4. CONCLISIONS

Carbon fibers could be deposited uniformly with duplex coatings of nickel (the internal layer) and cupper (the external layer) using an electroplating process. The thickness of Ni and Cu was about 1-2 ㎛. The surface fluorination of Cu-Ni-C fibers was carried out at 25-100℃ and 101 kPa for 1h. From the XPS results, the CuO layer on Cu film was decreased by surface fluorination at 25℃, and the CuF_2 was prepared by surface fluorination at 100℃. These surface modifications with fluorination caused to the increasing of surface roughness. Consequently, the adhesion between fluorinated Ni-Cu-C fibers and Mg alloys could be improved, and it led to the improvement of tensile strength of carbon fibers reinforced magnesium matrix composites

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