Loose porous composites of Ni/short carbon fibers prepared by electrodeposition

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Carbon fibers (CFs) are widely used due to the high specific strength, specific modulus, low expansion coefficient, electrical conductivity and relative flexibility [1–3]. Nowadays research into functional material applications of CFs has been actively pursued. The properties of these materials, such as electrical conductivity, expansion coefficient and mechanical properties can be improved by adding good conductive composites of meta/CFs. The composites of metal-coated three-dimensional braided CFs are developed, however, which is limited by high cost and complicated manufacturing process [4, 5].

Electrodeposition is a simple and utility method of codepositing particles, fibers or whiskers in the plated layer to fabricate metal matrix composites (MMCs) [6–8]. The present authors obtained the loose porous composites of nickel and short CFs by the similar technique. To our knowledge, this is the first report about the preparation of the porous metallic composites through the simple technology of electrodeposition. The loose porous structure offers innovative properties of Ni/short CFs composites, such as light density, high porosity and large specific area, thus the composites are expected to be promising functional materials, especially as three-dimensional reticulated conductive fillers applied in resins or rubbers. And the loose porous electrodeposits are prospected precursors for bulk materials of Ni alloy/short CFs using special casting techniques or powder metallurgy techniques.

Short CFs were manufactured in Shanghai Xinxing Carbon Co., Ltd in China. The Polyacrylonitrile (PAN)based CFs were desized after being dispersed, and were cut into 2–3 mm in length. These PAN-based CFs have a density of 1.76 g/cm³ and a mean diameter of 7 μ m. Prior to use, any contaminants were cleaned off from the fiber surfaces by immersing them into the acetone solution for about 40 min.

The Ni-plating bath (400 g/l Ni(SO₃NH₂)₂·4H₂O, 5 g/l nickel chloride, 40 g/l boric acid) was used. The concentration of short CFs in bath is 2 g/l. Before being plated, in order to enhance the interfacial adhesion, the short CFs were activated in nitric acid for about 10 min, and then mixed with 0.1 g/l surfactant (Hexadecyltrimethylammonium bromde), and at last agitated to uniformity by ultrasonic wave. A pure Cu plate with a small exposed surface of 1 cm² (0.2 cm × 5 cm) was used as the cathode. A pure Ni plate (99.98 wt.%) was used as the anode. The experiments were operated at $50 \degree$ C with slight agitation.

The density of the electrodeposits was measured using the Archimedes method with distilled water as the immersion medium. The content of short CFs in the electrodeposits was determined gravimetrically. The morphology of the short CFs and the separated electrodeposits were observed by scanning electron microscopy (SEM: S520) and digital camera (Canon Digital IXY500).

For other parameters in controlled conditions, two electrodeposits were prepared by using 5 and 10 $A \cdot dm^{-2}$ for 30 min, whose morphologies are shown in Fig. 1a and b. The densities of the two electrodeposits are 1.45 and 3.79 g/cm³, and the volume fractions of CFs are 47.6 and 8.3 vol.%, respectively. The images show that each Ni-coated CF laps the next and thus forms a loose porous structure. In the same processing time various thicknesses of Ni-coated short CFs were also obtained at various current densities. The average diameter of Ni-coated short CFs is about 10 μ m (Fig. 1a), while the other is about 40 μ m (Fig. 1b). Fig. 1c and d are the enlarged images taken by digital camera corresponding to (a) and (b), in which the electrodeposits are similar to candy-floss or villus.

It is well-known that CFs are good conductors. If only the short CFs are buried on the substrate or lapped others, which means they turn into an extended cathode, they will be coated by Ni like the substrate. In the Ni-plating bath, short CFs contact the substrate with a certain angle during impacting time. So CFs at the bottom are most probably protruding out of Ni coating on the substrate. Fig. 2 is a typical image about the situation on the substrate at the intermediate stage of electrodeposition (at 5 A·dm⁻², 5 min). It shows short CFs bury in Ni coating (shown by arrow A) and twist with others (shown by arrow B).

As the processing time increased, the protrudent ends of short CFs enlarged the contact area with the suspending short CFs in the bath, thus most of the short CFs migrated to the cathode and lapped others. Ni coated the lapped CFs and built up the loose porous structure. Fig. 3 is the SEM morphology of such structure, which is from the same electrodeposit prepared at 10 A·dm⁻²

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Figure 1 SEM images of two electrodeposits prepared at: (a) $5 \text{ A} \cdot \text{dm}^{-2}$, 30 min (b) 10 $\text{A} \cdot \text{dm}^{-2}$, 30 min. Enlarged images corresponding to (a), (b) are shown in (c), (d).



Figure 2 SEM image of the buried short CFs and some of them lapped together (5 $A \cdot dm^{-2}$, 5 min).

for 30 min. It could be seen in Fig. 3 that the lapped short CFs are an integer by Ni coatings.

However, due to the various influencing factors, local electrodeposits are not uniform in some parts of the specimens. There exit sparse and dense parts of Ni-coated CFs in the two electrodeposits as shown in Fig. 1. And in the same electrodeposit prepared at 10 A·dm⁻² for 30 min, we observed two lapped short CFs with different thickness Ni coatings, as shown in Fig. 4. The diameters of the two short CFs are about 45 and 25 μ m, respectively. The difference in diameter may be attributed to the insufficient interac-



Figure 3 SEM morphology of the lapped short CFs (10 $A \cdot dm^{-2}$, 30 min).

tion time or the poor contact between these Ni-coated fibers.

In summary, the loose porous composites of Ni/short CFs were successfully prepared by electrodeposition. The densities of the two composites are 1.45 and 3.79 g/cm³, and the CFs contents are 47.6 and 8.3 vol.%, respectively. The electrodeposits are distinct in configuration from other composite coatings. The former has the three-dimensional reticulated structure and a peculiar candy-floss shape. However, the electrodeposits are non-uniform in local. After all, it is expected that, based on the results of the present study, a more homogeneous



Figure 4 SEM morphology of the lapped short CFs with different thickness Ni coatings ($10 \text{ A} \cdot \text{dm}^{-2}$, 30 min).

and regular porous composite can be fabricated by improving the process.

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