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Fabrication of superaligned carbon nanotubes reinforced copper matrix laminar composite by electrodeposition

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Abstract: A new kind of laminar metal matrix nanocomposite (MMC) was fabricated by an electrodeposition process with copper and superaligned carbon nanotubes film (SACNT film). The SACNT film was put on a titanium plate and then a layer of copper was electrodeposited on it. By repeating the above process, the laminar Cu/SACNT composite which contains dozens or hundreds of layers of copper and SACNT films was obtained. The thickness of a single copper layer was controlled by adjusting the process parameter easily and the thinnest layer is less than 2 μm. The microscopic observation shows that the directional alignment structure of SACNT is retained in the composite perfectly. The mechanical and electrical properties testing results show that the tensile and yield strengths of composites are improved obviously compared with those of pure copper, and the high conductivity is retained. This technology is a potential method to make applicable MMC which characterizes high volume fraction and directional alignment of carbon nanotubes.

Key words: copper matrix laminar composite; superaligned carbon nanotubes; electrodeposition

1 Introduction

Copper is widely used in manufacturing of electric equipments and heat conductors due to its high electrical and thermal conductivity. The use of pure copper, however, is limited by its low strength and high density.

The traditional methods to improve the mechanical properties of pure copper, such as alloying or dispersing with second phase, have to bear the cost of decreased performance in electrical and thermal conductivity. The previous studies have made various attempts to solve this problem [1−3]. LU et al [4] synthesized pure copper samples with a high density of nanoscale growth twins which showed a tensile strength more than 1 GPa, while retaining an electrical conductivity comparable with that of pure copper. WEI et al [5] prepared a Cu−0.5%Cr alloy after the processing of four passes of equal channel angular pressing (ECAP) followed by 90% cold rolling and aging at 450 °C for 1 h, obtaining the tensile strength of 554 MPa, elongation to failure of 22% and electrical conductivity of 84% IACS [5]. Nevertheless, these materials, which show excellent performances in mechanical and electrical properties, cannot be applied in practical application in most cases because of their small size and complex processes.

Recently, a so-called “composite method” has made successful attempt to prepare copper matrix composites that are composed of two or more kinds of materials by a certain technological process. In a variety of micro-materials and nanomaterials, carbon nanotube is an ideal reinforcement material because of its excellent mechanical, electrical, and thermal properties [6−10].

Most of the previous studies on the Cu/carbon nanotubes (CNT) composites focused on how to improve the mechanical properties of copper matrix, do not address the conductive performance of the copper matrix [11−16]. Furthermore, the CNTs in these composites are dispersed in all directions randomly. If CNTs are lined up in the same direction within the copper matrix, they will enhance the mechanical and electrical properties in one particular direction.

Superaligned carbon nanotubes (SACNT) film [17] is a uniform CNT film processed from superaligned CNT arrays, which is provided by Tsinghua−Foxconn Nanotechnology Research Center. The SEM image of

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SACNT film is shown in Fig. 1 [18]. These superaligned CNT arrays were first synthesized on silicon wafers using chemical vapor deposition (CVD) in 2002 [19]. Compared with the traditional CNT films, the carbon nanotubes in SACNT film are all lined up in the same direction. This characteristic endows the SACNT film with particular advantages to improve the material properties in one direction and enhance concerned properties of materials with the SACNT films.

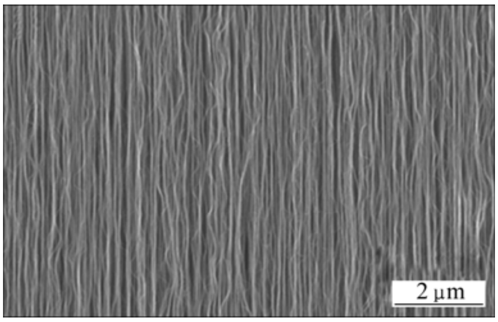

Fig. 1 SEM image of SACNT film

The SACNT has been used to prepare CNT/ polyvinyl alcohol (PVA) composite yarns [20] and CNT/ epoxy composites [21,22], and to our best knowledge, it has never been used to prepare metal matrix composites.

Some previous studies have reported several methods to prepare high strength copper [4,11,14,23]. However, the small size of these materials and the complex process limit the practical application of them. In this study, the samples with demensions of 180 mm × 55 mm × 0.1 mm were prepared. Since the SACNT films can be pulled out continuously and the copper layers can be accumulated endlessly, this composite can be expanded to any size theoretically.

2 Experimental

2.1 Laminar Cu/SACNT composite prepared by electrodeposition process

A layer of SACNT film was pulled out from an SACNT array on an 8-inch wafer. The SACNT film should be wider than 55 mm so that it can completely cover the surface of the titanium plate, as described later.

A piece of titanium plate (size: 180 mm × 55 mm × 1 mm) was used as the cathode plate. In order to "fix" the aforementioned SACNT film to the surface of the titanium plate, ethanol was uniformly applied to the surface and then dried out completely. The other side of the titanium plate was pasted with a layer of electrical tape to avoid the copper deposition.

A piece of phosphor copper plate (size: 180 mm × 55 mm × 2 mm) was used as the anode plate. Similarly, one side of the plate was pasted with a layer of electrical tape.

The cathode plate and the anode plate were merged into an electrolytic bath (sizes: 400 mm × 150 mm × 250 mm) with a spacing of 300 mm between the two plates. The cathode plate and anode plate were connected to the negative and positive electrodes of a DC power, respectively, as shown in Fig. 2.

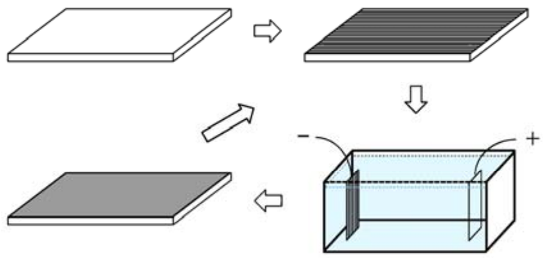

Fig. 2 Schematic of electrodeposition process

The electrodeposition process was performed in an electrolyte of $CuSO_4 \cdot 5H_2O$ (300 g/L), H_2SO_4 (50 g/L), and glucose (5 g/L) at room temperature. The electric current density (0.03 A/cm^2) and the time (2, 4 or 8 min) determine the thickness of coating to cover the SACNT film. The coating covering the SACNT film formed a new surface. A subsequent layer of SACNT film was placed on the new surface. By repeating all these processes several times, a piece of laminar Cu/SACNT composite was prepared on the surface of the titanium plate. These steps are illustrated in Fig. 2.

Titanium is an active metal, and it is very easy to strip laminar Cu/SACNT composite from the surface of titanium plate because it is attacked by oxygen to form an adherent oxide layer.

The typical mass per unit area of the SACNT film is 1.5 $\mu g/cm^2$ [18]. The volume fraction of the SACNT film in the composite can be calculated by

$$\varphi_s = m_s\rho_c/[m_s\rho_c+(m-m_s)\rho_s] \quad (1)$$

$$m_s = 1.5S \quad (2)$$

where φ_s stands for the volume fraction of the SACNT film, m_s is the mass of the SACNT film in the sample of composite, m signifies the mass of the sample of composite, ρ_c denotes the density of copper, ρ_s is the density of multi-walled carbon nanotubes in the SACNT film, and S stands for the area of the sample.

In order to explore the effect of different volume fractions of SACNT film on the composites' mechanical and electrical properties, the composites with three different volume fractions of SACNT film and a sample without SACNT film were prepared.

When the three kinds of composites were prepared, the electric current densities were identical. Therefore, it is the electrodeposition time that determines the plating thickness. With decreasing the thickness of copper layers, the volume fraction of SACNT increases accordingly.

For composites of 1.3%, 2.6%, and 5.2% SACNT (volume fraction), the thicknesses of the copper layers are approximately 4, 2 and 1 μm, respectively. The copper layers and SACNT films desire geometric shapes, such as uniform thickness, smooth interface and uniform directionality.

Table 1 Process parameters of pure copper and laminar Cu/SACNT composites

Volume fraction of SACNT film/%	Number of copper layers	Number of SACNT film layers	Electro-deposition time of every copper layer/min	Total electro-deposition time/min
0	1	0	180	180
1.3	20	19	8	160
2.6	40	39	4	160
5.2	60	59	2	120

2.2 Preparing samples for microscopic observation

The samples were taken from the laminar Cu/SACNT composites. Each sample had two orthogonal cross-sections: one was parallel to the extension direction of the SACNT film, the other was perpendicular to the direction, as shown in Fig. 3. We named them parallel cross-section and perpendicular cross-section for short. The cross-section view was prepared in a resin mounted and polished sample.

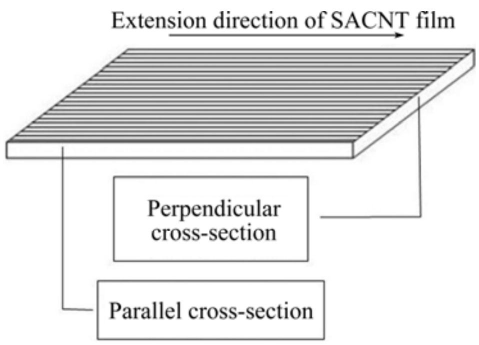

Fig. 3 Schematic of parallel cross-section and perpendicular cross-section

2.3 Mechanical and electrical properties testing

Mechanical testing was accomplished in an Instron 5848 tensile tester. Each tensile specimen was 70 mm in length and 5 mm in width, and the thickness was kept the same as that of the aforementioned samples.

The electrical testing was accomplished in an SB2230 precision digital resistor. The pure copper and three kinds of composites were made into elongated samples, which were 120−150 mm in length and 10−14 mm in width. With this precision digital resister, a four-circuit measuring method was employed to measure the resistance of all the four samples. The electrical resistivity and conductivity were calculated with the resistance and size of the samples.

The above specimens for mechanical testing and electrical testing were parallel to the direction of CNTs in their lengthwise direction. This direction was named “longitudinal direction”. In order to study the impact of the direction of CNTs on the properties, the specimens that were perpendicular to the direction of CNTs were also tested. This direction was named “lateral direction”. Every specimen in the lateral direction had a size of 50 mm in length and 5 mm in width and the thickness was the original one from the sample.

3 Results and discussion

3.1 Microstructure observation

The samples were first polished so that the copper could cover the surface uniformly. Figure 4(a) shows the metallograph of the polished cross-section before the corrosion processing to be mentioned later. The samples are then subjected to mild corrosion to remove the copper on the SACNT films to observe their internal structures. The corrosion was performed in an ethanol solution of HCl (10 g/L) and $FeCl_3$ (30 g/L) within 3 s.

In Figs. 4(b)−(g), the white parts are actually the copper layers and the dark lines are the SACNT films. Since the CNTs in the SACNT films are all lined up in the same direction, the zero dimensional form of CNTs in the perpendicular cross-section and one dimensional form of CNTs in the parallel cross section can be observed in the SEM images. Comparing Figs. 4(b), (d) and (f) with Figs. 4(c), (e) and (g), one can spot differences between the perpendicular cross-section and the parallel cross-section. The black lines in the perpendicular cross-section are wave-shaped and discrete, which indicates that CNTs in the SACNT films are not strictly aligned in a straight line. Furthermore, the shape of each waved line is similar to that of the adjacent lines. The “heredity” of waved shape comes from the waved plating surface. On one hand, it is hard for an electrodeposition process to produce a very flat surface at this scale. On the other hand, the uneven surface influences the shape of next layer of the copper and SACNT film. Unlike the perpendicular cross-section, black lines in the parallel cross-section are straighter and longer. This is due to the fact that the SACNT films maintain straight-line shape with the tension along the length of CNTs. The tension comes from the process of pulling out the SACNT film from the SACNT array.

Figures 5(a)−(c) show the SEM images of Cu−2.6%SACNT composite, which provide more details of the shape, distribution and directionality of CNTs and the interface between the CNTs and copper. Figure 5(a) shows that the ends of the CNTs look like some white

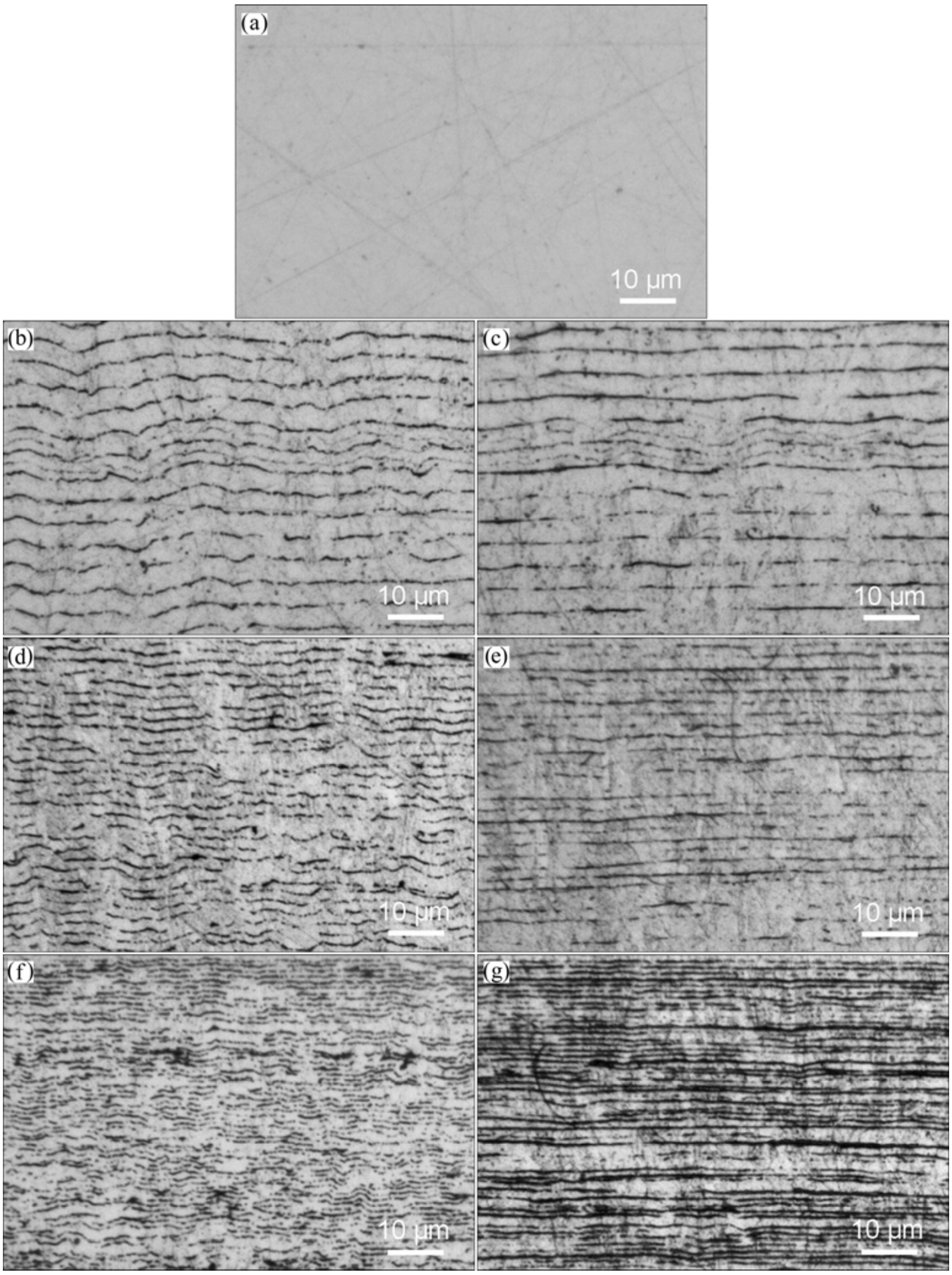

Fig. 4 Metallographs of polished cross-section before corrosion processing (a), and polished and corroded cross-section of Cu−1.3%SACNT (b, c), Cu−2.6%SACNT (d, e) and Cu−5.2%SACNT (f, g)

dots. These CNTs have been cut off by the polishing process, because they are perpendicular to this plane in the composite. In the same way, the CNTs in Fig. 5(b) are parallel to the plane of sample for microscopic observation so that the whole CNTs can be observed. Figure 5(c) shows the fracture of the tensile test sample. The CNTs within the copper matrix have been pulled out from the fracture sequentially. Figure 5 indicates that the SACNT films maintain their original superaligned form in the composite during the electrodeposition process. The CNTs are dispersed homogeneously between two layers of the copper and are lined up in the same direction. However, some small gaps around the CNTs can be spotted, which imply the gaps in the SACNT films cannot be filled with copper completely by this traditional copper sulfate electrodeposition process. This is a problem that needs to be solved in future.

In order to explore the interface between CNTs and copper in atomic scale, TEM images were taken. Figure 6(a) shows several independent CNTs in the copper matrix. The similar characteristics of CNTs confirm the conclusions drawn from the SEM images. Figure 6(b) shows the high resolution TEM (HRTEM) image. The upper half shows the typical hexagonal

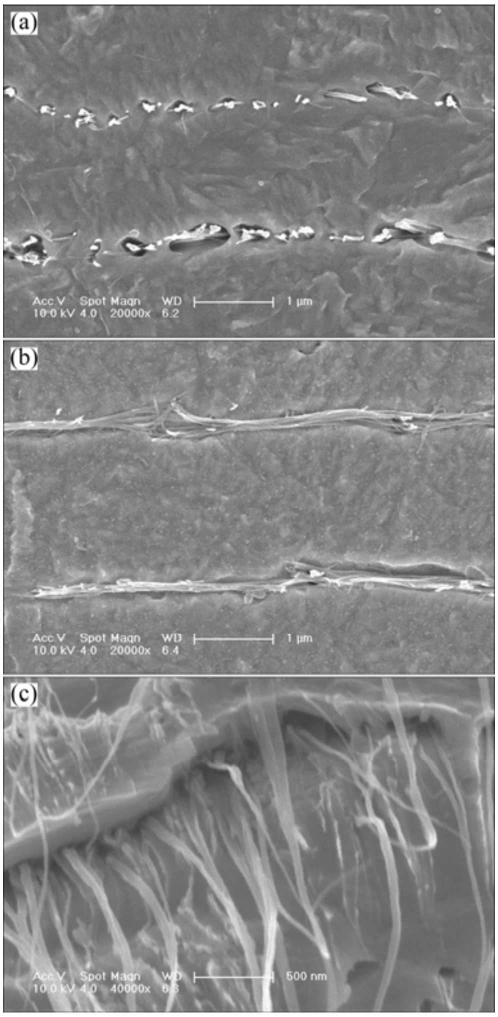

Fig. 5 SEM images of Cu−2.6%SACNT composite of perpendicular cross-section (a) and parallel cross-section (b), and fracture of tensile test sample (c)

structure of copper. The bottom half shows the irregular wavy stripes which represent the structure of multi-walled carbon nanotube. The bonding at the interface between CNT and copper is well, which will help improving the mechanical properties of matrix by the way of load transfer.

Figure 7 shows the Raman spectra of laminar Cu/SACNT composites. The composites with different volume fractions of SACNT have the same *D*-peak (1327 cm^{-1}) and *G*-peak (1584 cm^{-1}) which are characteristic of CNTs.

3.2 Mechanical and electrical properties

Figure 8 shows the average yield strengths, tensile strengths, and conductivities of pure copper and Cu/SACNT composites in the longitudinal direction. The samples of pure copper and composites were prepared by the same method. Accordingly, the only difference,

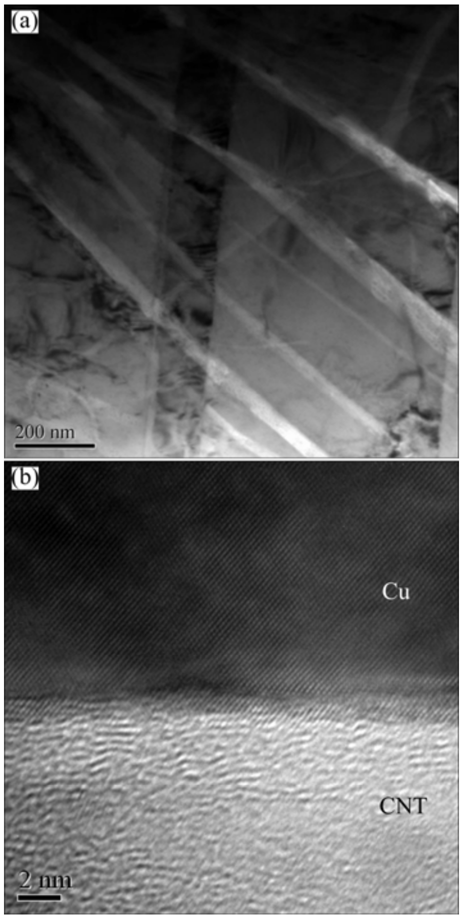

Fig. 6 TEM (a) and HRTEM (b) images of Cu−2.6% SACNT composite

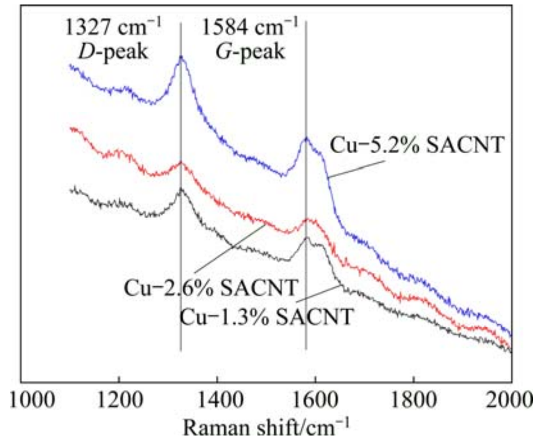

Fig. 7 Raman spectra of laminar Cu/SACNT composites

between these samples is the volume fraction of CNTs which influences the mechanical and electrical properties.

The average yield and tensile strengths of pure copper are 80 and 197 MPa, respectively. As the volume fraction of CNTs increases, the average tensile strength of composite increases. The average tensile strengths of composites with 1.3% SACNT, 2.6% SACNT and 5.2%

SACNT are 243, 272 and 290 MPa, respectively. The increase of average yield strength (0.2% offset strain) is more significant than that of tensile strength. The average yield strengths of composites with 1.3% SACNT, 2.6% SACNT, and 5.2% SACNT are 130, 145 and 160 MPa, respectively.

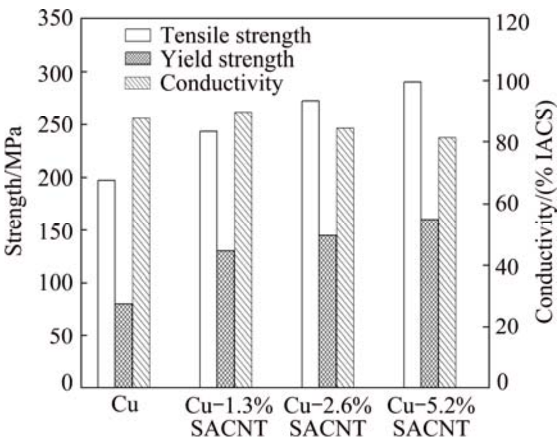

Fig. 8 Mechanical and electrical properties of Cu and Cu/SACNT composites in longitudinal direction

The conductivity of pure copper is 87.7% IACS. The presence of SACNT films has a much less effect on the conductivity than on the strength. The conductivities of composites are 89.7% IACS (1.3% SACNT), 84.5% IACS (2.6% SACNT) and 81.4% IACS (5.2% SACNT). When the volume fraction of CNTs is low, the Cu/SACNT composite has the same conductivity with the pure copper. Two reasons lie behind the fact that higher volume fraction of CNTs leads to slight degradation in conductivity. Firstly, the conductivity of CNTs is not as good as that of pure copper. Secondly, the small gaps around the CNTs, as shown in Fig. 5(a), have adverse effects on the conductivity.

Figure 9 shows the representative stress−strain curves of Cu and Cu/SACNT composites. The pure copper has good plasticity at room temperature. The maximum strain for pure copper is 23.5%, and the maximum strain decreases with increasing the volume fraction of SACNT. For the composites with 1.3% SACNT, 2.6%SACNT and 5.2%SACNT, the maximum strains are 14.5%, 13.0% and 8.0%, respectively. Even so, the plasticity of these composites is quite satisfactory for secondary processing. Furthermore, the annealing process is helpful to secondary processing that needs large deformation.

Figure 10 compares the properties of Cu/SACNT composites in the longitudinal direction and lateral direction. Compared with the tensile strengths and conductivities in the longitudinal direction, those in the

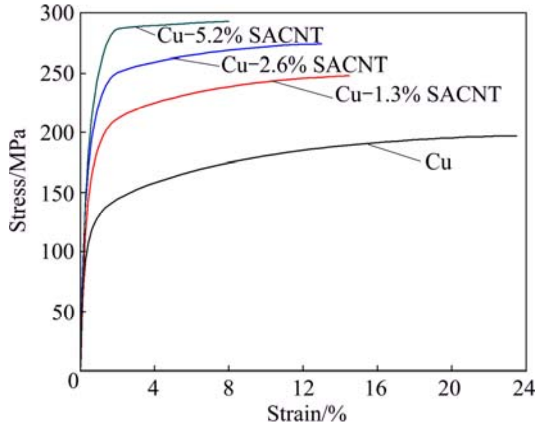

Fig. 9 Representative stress−strain curves of Cu and Cu/SACNT composites

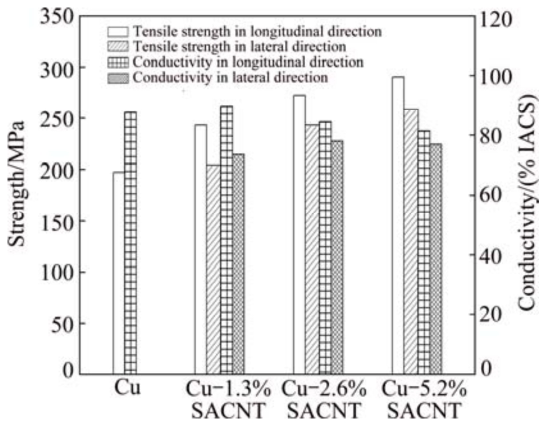

Fig. 10 Mechanical and electrical properties of Cu and Cu/SACNT composites in longitudinal direction and lateral direction

lateral direction are significantly lower. The tensile strengths in the lateral direction are 204 (1.3% SACNT), 243 (2.6% SACNT) and 257 MPa (5.2% SACNT). In contrast, those in the longitudinal direction are 243 (1.3%SACNT), 272 (2.6%SACNT), and 290 MPa (5.2%SACNT). The conductivities in the lateral direction are 73.5% IACS (1.3% SACNT), 78.0% IACS (2.6% SACNT) and 76.8% IACS (5.2% SACNT). In contrast, those in the longitudinal direction are 89.7% IACS (1.3% SACNT), 84.5% IACS (2.6% SACNT), and 81.4% IACS (5.2% SACNT). Figure 10 shows that the strengthening effect of SACNT films in the lateral direction is much lower than that in the longitudinal direction.

It can be also seen from Fig. 10 that the tensile strengths in the lateral direction show a growth trend similar to that in the longitudinal direction. This is because the directional alignment structure of SACNT films is never ideal, as there is always a small portion of CNTs aligned in random directions.

4 Conclusions

1) Electrodeposition is a simple and effective process to prepare large-sized laminar Cu/SACNT composites. The directional alignment structure of SACNT is retained in the composite perfectly. This characteristic allows CNTs to play a role in reinforcing tensile strength and conductivity in the aligned direction of CNTs.

2) Compared with the pure copper, the mechanical properties of laminar Cu/SACNT composites are significantly improved. With increasing the volume fraction of SACNT films, the mechanical properties of composites are improved more and more. In the meanwhile, the presence of SACNT films has little impact on the conductivity.

3) The laminar Cu/SACNT composites show anisotropic characteristics, which is caused by the direction of CNTs. The tensile strength and conductivity in the lateral direction are lower than those in the longitudinal direction.

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电沉积法制备超顺排碳纳米管增强铜基层状复合材料

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摘 要：采用电沉积法，使用纯铜和超顺排碳纳米管薄膜(SACNT)制备新型金属基复合材料。将 SACNT 铺放在钛板上，然后在其表面电沉积一层纯铜。通过不断重复铺膜和电沉积的过程，可以得到含有几十层到上百层碳纳米管薄膜的铜基层状复合材料。通过调整电沉积参数可以控制电沉积的每一层纯铜的厚度，每一层纯铜的厚度可以小于 2 μm。通过显微组织分析发现，SACNT 的超顺排特性在复合材料中得到保留。力学性能和电学性能测试结果表明：相比于纯铜材料，复合材料的抗拉强度和屈服强度均大幅提升，且导电能力没有受到明显影响。这种工艺可以大批量制备含有高体积分数和定向排布特征碳纳米管的金属基纳米复合材料，具有良好的实际应用前景。

关键词：铜基层状复合材料；超顺排碳纳米管；电沉积

(Edited by Mu-lan QING)