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ARTICLE

Tensile Properties and Fracture Mechanism of Copper Matrix Composites Reinforced by Carbon Fibres with Ni-Co-P Alloy Coating

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Abstract: The composition controlled Ni-Co-P multi-component alloy coatings with the thickness of about 0.7 μm were prepared on the surface of carbon fibres (CFs) using the palladium-free electroless plating. Then the copper matrix composites reinforced by the Ni-Co-P coated CFs, namely the CF/Cu(Ni-Co-P) composites, with CF content of 10vol%, 20vol%, 30vol%, and 40vol%, the uncoated CFs reinforced copper matrix composites (CF/Cu) with 30vol% CFs, and the Ni-coated CFs reinforced composites, namely the CF/Cu(Ni) composites, with 30vol% CFs were prepared by the vacuum heat pressure sintering at 850 °C and 30 MPa. The distribution of reinforcement and interface phase elements and the fracture morphologies of the composites were observed by the scanning electron microscope and energy dispersive spectrometer. The tensile properties of different copper matrix composites were characterized by the electronic universal testing machine. Results show that the smooth Ni-Co-P coatings of uniform thickness can be obtained on CFs surface by electroless plating at 70 °C for 10 min. The tensile properties of CF/Cu(Ni-Co-P) composites are firstly increased and subsequently decreased with increasing the CFs content. The highest tensile and yield strengths are obtained for the 30vol% CF/Cu(Ni-Co-P) composite. The mechanical properties of CF/Cu(Ni-Co-P) composite are obviously better than those of the CF/Cu and CF/Cu(Ni) composites with the same content of reinforcement (30vol%). The fracture mechanism of CF/Cu(Ni-Co-P) composite is non-accumulative fracture.

Key words: Ni-Co-P coated carbon fibres; copper matrix composites; tensile properties; fracture mechanism

With excellent tensile strength, high stiffness and modulus, low coefficient of thermal expansion, and low density[1-3], the carbon fibres (CFs) have the great potential application to improve the mechanical properties of metal matrix composites as the reinforcement[4]. CFs reinforced copper matrix (CF/Cu) composites have the advantages of good electrical and thermal conductivity (due to Cu) and excellent mechanical properties (due to CFs), and display superior mechanical and physical properties, including the low coefficient of thermal expansion, high specific strength and modulus, and good wear resistance, therefore attracting widespread attention in the manufacture of integrated structure/function materials, such as electrical brushes and pantograph slides[5,6]. However, the mechanical properties of CF/Cu composites are worse than their theoretical values because of the imperfect load transfer effect, which is caused by the poor wettability and the absence of interfacial reaction between CFs and Cu matrix[7,8]. Thus, improving the interfacial wettability and interfacial bonding strength between CFs reinforcement and Cu matrix is important for the property enhancement of the CF/Cu composites[5,7,9-12].

The CF/Cu composites prepared by sintering and electroplating methods exhibit the better defect-free interface, compared with the ones prepared by the mixing and sintering methods. However, the weak mechanical interface bonding is barely ameliorated with the preparation method

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change[8]. In order to enhance the interfacial bonding strength, Veillere et al[13] studied the effects of matrix alloying with chromium on the interface type and bonding nature, and found that the bonding strength is increased and the interface bonding type changes from mechanical bonding to metallurgical bonding due to the formation of chromium carbide (Cr_3C_2) interphase at the interface. Whereas, the reaction between CFs and chromium results in the graphitization and the strength reduction of CFs, thereby leading to the ripping shape of the fracture of CFs. Yang et al[6] found that the titanium and nickel coatings deposited on CFs surface can effectively improve the interface bonding strength between CFs and Cu matrix and avoid the pore defects. It is worth noting that Ni is the optimal candidate for interface modification of C/Cu composites, because it can not only dissolve into Cu infinitely[14], but also has the fine wettability with carbon. Compared with other metal elements, Ni has obviously better wettability, owing to the Ni-C-O bonds formed between CFs and Ni[15] through the chemical solution deposition method. Ni alloy coatings not only increase the density of C/Cu composites, but also change the crack propagation mechanism[16,17], which indirectly enhances the load-bearing capacity of composites. Unfortunately, the graphitization of CFs caused by the diffusion-precipitation between Ni and C during sintering process of composites may decrease the tensile strength of CFs. Dong et al[18] studied the effect of Ni-Cu duplex alloy coatings on the friction and wear resistance of CF/Al composites, and found that CF graphitization induced by Ni can be restrained due to the presence of NiAl compound, which hinders the formation of Cu-Ni solid solution, crack initiation, and crack propagation at the interface. Currently, the effects of Al-*M* alloy coatings on interface microstructure of CF/Cu composites are rarely reported.

Co has excellent wettability and compatibility with CFs[19] and may restrain the CF graphitization through the alloying on nickel coatings. In this research, the nickel-cobalt-phosphorus (Ni-Co-P) coated CFs were prepared by palladium-free electroless plating, and then the Cu matrix composites with different contents of Ni-Co-P coated CFs and uncoated CFs were synthesized by vacuum hot pressing sintering. By the comparison of stress-strain curves of the copper matrix composites reinforced by Ni-Co-P coated CFs, namely CF/Cu(Ni-Co-P) composites, the copper matrix composites reinforced by uncoated CFs, namely CF/Cu composites, and the copper matrix composites reinforced by Ni coated CFs, namely CF/Cu(Ni) composites, the influence of Ni-Co-P coatings and CFs on tensile properties of Cu matrix composites was analyzed. In addition, the strengthening effect of reinforcement and fracture mechanism of the composites were studied.

1 Experiment

The essential parameters of original CFs (Gansu Haoshi Carbon Fibre Co., Ltd, Gansu, China) are shown in Table 1. The composition of the electroless plating solution for Ni-Co-P coating is listed in Table 2, and all chemical reagents used in the electroless plating process are analytically pure.

Fig. 1 shows the preparation process of Ni-Co-P coating on CF surface. Firstly, the initial CFs were immersed in 68wt% HNO_3 at 90 °C for 30 min, subsequently washed several times by the distilled water, and then dried in thermostatic drying chamber at 60 °C for 6 h. Secondly, the CFs fixed on the square framework were immersed in 2 mol/L $NiSO_4$ solution for 10 min, and then dried at 100 °C for 4 h. Thirdly, the activated CFs washed by distilled water were reduced in the reduced aqueous solution containing 20 g/L KBH_4 and 50 g/L NaOH at ambient temperature for 10 min, and were subsequently rinsed by the distilled water to clean the residual reductant. Finally, the CFs after activation-reduction were immersed in Ni-Co-P electroless plating solution at 70 °C for 10 min, and then rinsed and dried at 40 °C for 8 h using the vacuum drying oven.

As shown in Fig. 2, the CF/Cu(Ni-Co-P) composites were firstly fabricated through the repetitive overlapping of copper foil with the thickness of 0.01 mm and Ni-Co-P coated CFs. The refined copper foil was cut into the foils of 12 mm×50 mm, and the Ni-Co-P coated CFs were uniformly dispersed into the lamellar structure. Subsequently, the lamellar fibres and Cu foil were placed layer by layer in the graphite mold. The CFs content was controlled as 10vol%, 20vol%, 30vol%, and 40vol% in the composites. Ultimately, the CF/Cu composites were sintered by vacuum hot pressing at 850 °C and 30 MPa for 40 min. The same process was used to prepare the CF/Cu composites with 30vol% CFs and the CF/Cu(Ni) composites with 30vol% CFs.

X-ray diffraction (XRD) analysis of phase composition of Ni-Co-P coatings was conducted by the D/max 2400 XRD with the monochromatized Cu Kα radiation (λ=0.154 178 nm) at a scanning speed of 5°/min. The microstructure and element content of Ni-Co-P coated CFs and the distribution of fibres and interfacial elements of Cu matrix composites were investigated by the transmission electron microscope (TEM) and the scanning electron microscope (SEM, Quanta FEG-450 SEM) coupled with the energy dispersive spectrometer (EDS). The tensile tests for composites were conducted by the electronic universal testing machine (MTS-E44.304, USA) with a constant tensile rate of 0.2 mm/min at ambient temperature. Before the tensile test, the Cu matrix composites were firstly processed into the tensile specimens with a gauge distance of 15 mm by the electrical discharge machining, and were subsequently polished with 2000# waterproof abrasive paper. The fracture morphologies of the composites were

Table 1 Essential parameters of CFs

Specification	Diameter, D/μm	Elastic modulus, E/GPa	Elongation, δ/%	Density, ρ/g·cm^{-3}	Carbon content/wt%
PAN-T300	~7	206	1.5	1.76	98.5

Table 2 Composition and functions of electroless plating solution for Ni-Co-P alloy coating

Component	Content/g·L^{-1}	Function
$NiSO_4·6H_2O$	13.15	Main salt
$CoSO_4·7H_2O$	14.05	Main salt
$C_6H_5Na_3O_7$	40.00	Complexing agent
$(NH_4)_2SO_4$	40.00	Buffer
NaH_2PO_2	20.00	Reducing agent
NaOH	-	pH adjustment

observed by SEM.

2 Results and Discussion

2.1 XRD analysis of CFs before and after modification

XRD patterns of CFs with and without Ni-Co-P coatings are shown in Fig.3. It can be seen that the uncoated CFs have two diffraction peaks at 2θ=25.8° and 42.6°, corresponding to the (002) and (100) crystal planes of amorphous carbon, respectively. Moreover, some small diffraction peaks appear, corresponding to the graphitization crystallization of the trace amount of carbon. Compared with the uncoated CFs, the Ni-Co-P coated CFs exhibit the crystallization peaks of Ni and Co with the face-centered cubic structure, and no diffraction peak of carbon exists[20]. Some diffraction peaks suggest that the P element exists in the form of nickel phosphorus compounds, such as Ni_7P_3 and Ni_2P phases, which are formed by the Ni-P codeposition. Besides, it is also confirmed that the coatings are fairly pure and have high crystallinity.

2.2 Morphology and deposition of Ni-Co-P coatings

SEM morphologies of CFs with and without Ni-Co-P coatings are shown in Fig.4. According to Fig.4a, a number of axial ditches appear on the surface of uncoated CFs after acid treatment, which is conducive to the adsorption of activated Ni^{2+} ions and can improve the mechanical bonding strength between the coatings and CFs. After deposition at 70 °C for 10 min, the smooth and consecutive Ni-Co-P coatings with a homogeneous thickness of ~0.7 μm are obtained on CFs surface (Fig. 4c). However, there are some cellular Ni-Co-P particles distributed on the coating surface. According to the EDS spectrum in Fig. 4c, the Ni content of the coating is 61.4wt%, which is significantly higher than the Co content (~28.9wt%) and P content (~9.7wt%). The microstructure and composition of Ni-Co-P coatings are determined by the differences between the Ni and Co deposition mechanisms. After the reduction by the KBH_4+NaOH mixed solution, the Ni^{2+} ions adsorbing on CFs surface are reduced to active Ni atoms (Ni^0). According to the atomic hydrogen theory[21], the active hydrogen H_{ad} derives from the reaction of sodium hypophosphite with the catalysis of Ni^0, as follows:

$$H_2PO_2^-+H_2O\xrightarrow{\text{Catalysis}}HPO_3^{2-}+2H_{ad}+H^+ \tag{1}$$

Ni^{2+} and Co^{2+} react with H_{ad} to form Ni and Co, as shown in Eq.(2) and Eq.(3), respectively:

$$Ni^{2+}+2H_{ad}\rightarrow Ni+2H^+ \tag{2}$$

$$Co^{2+}+2H_{ad}\rightarrow Co+2H^+ \tag{3}$$

The reaction rate of Eq. (2) is faster than that of Eq. (3) owing to the relatively high standard electrode potential of Ni ($E^\ominus$=−0.250 V), compared with that of Co ($E^\ominus$=−0.277 V)[22]. Thus, the deposition amount of Ni atoms is greater than that of Co atoms, i.e., the Ni-Co-P coatings are mainly composed of Ni and a small amount of Co and P[23]. Besides, the reduced Ni and Co atoms are not deposited on CFs surface. They gather together to form the atom clusters of different sizes at the solid-liquid interface[24,25] and subsequently to form the Ni-Co-P particles with chain shape, as shown in Fig. 4b. The Ni-Co-P particles with different sizes are formed in the initial

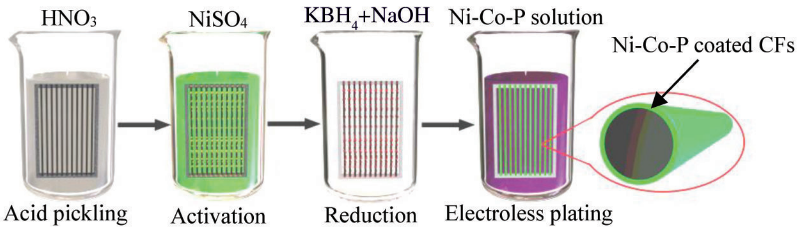

Fig.1 Preparation process of Ni-Co-P coating on CF surface

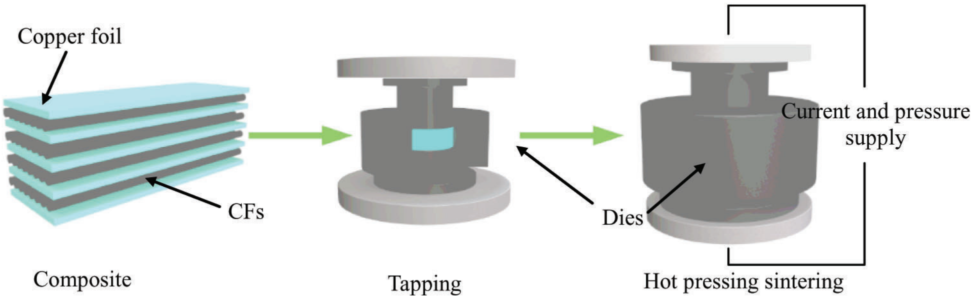

Fig.2 Preparation of CF/Cu composites

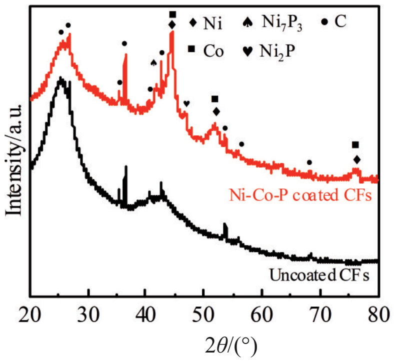

Fig.3 XRD patterns of uncoated and Ni-Co-P coated CFs

stage of deposition (3 min). After deposition for 10 min, the continuous Ni-Co-P coatings with trace cellular particles appear and cover the CFs surface.

2.3 Microstructure of CF/Cu(Ni-Co-P) composites

The microstructures of CF/Cu(Ni-Co-P) composites with 10vol%, 20vol%, 30vol%, and 40vol% CFs are shown in Fig.5. The CFs are uniformly distributed in the Cu matrix and show tight bonding with the Cu matrix. No agglomeration occurs when CFs content is below 30vol%. With increasing the CFs content to 40vol%, the homogenous distribution of CFs becomes worse and a few cavity defects occur at the interface caused by the excess addition of fibres, as shown in Fig.5d. The load transfer effect and the forming ability of Cu matrix are thereby worsened. In addition, Ni, Co, and P elements are accumulated on the interface between the fibres and Cu matrix owing to the non-diffusion effect of the Ni-Co-P coatings. This result indirectly indicates that the composite strength is unrelated to the alloying strengthening effects of the alloy coatings.

TEM microstructures of the interfaces in CF/Cu(Ni-Co-P) composites are shown in Fig.6. Fig.6b shows that there is an obvious boundary between CFs and coatings and no cavities occur, indicating the good interface bonding state. Besides, the microstructures of CFs near the interface show no significant

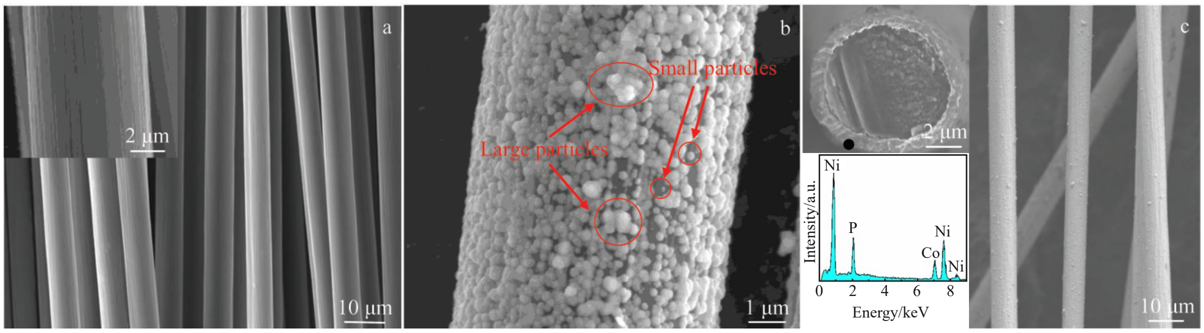

Fig.4 SEM morphologies of CFs without (a) and with Ni-Co-P coatings after deposition for 3 min (b) and 10 min (c)

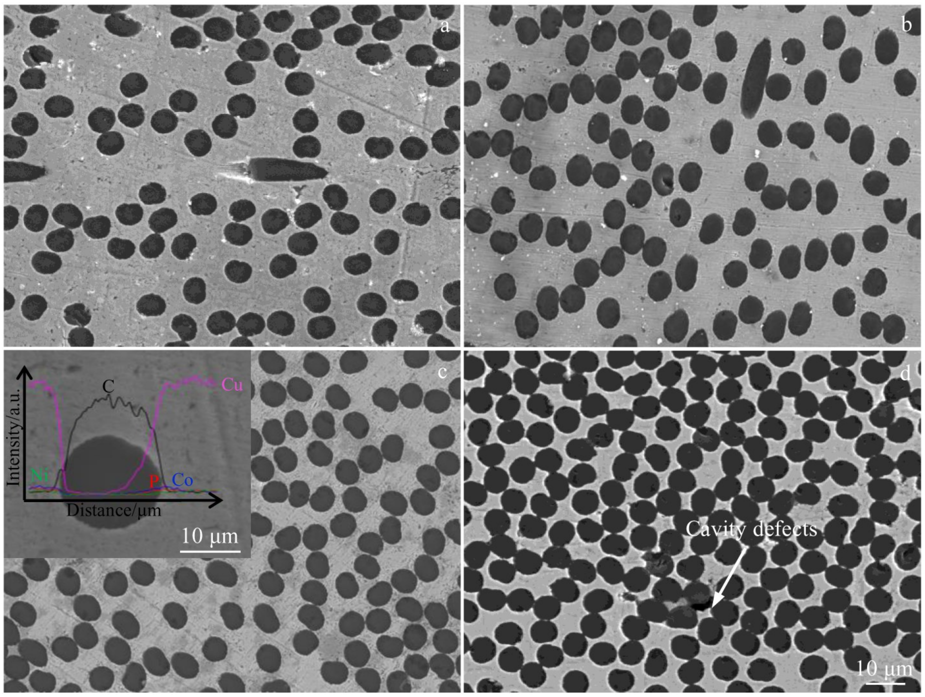

Fig.5 Microstructures of CF/Cu(Ni-Co-P) composites with 10vol% (a), 20vol% (b), 30vol% (c), and 40vol% (d) CFs

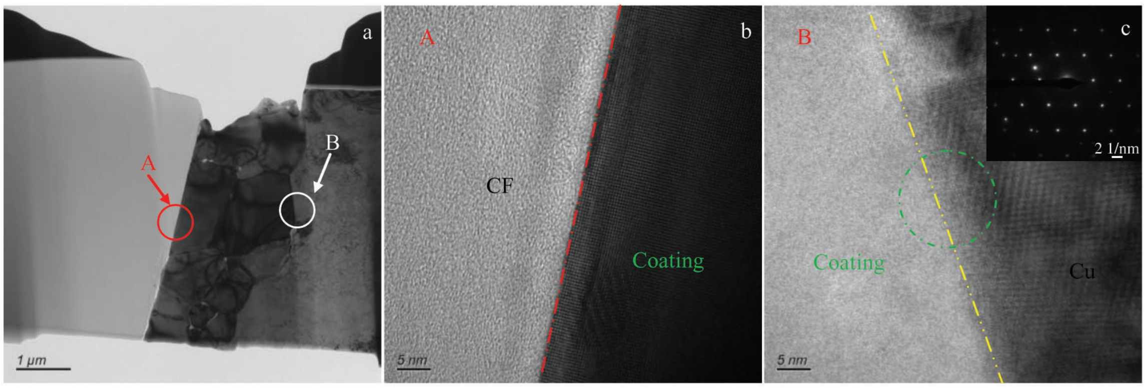

Fig.6 TEM microstructure of interfaces in CF/Cu(Ni-Co-P) composites (a); magnified TEM microstructures of area A (b) and area B (c) in Fig.6a

alteration, suggesting that the Ni-Co-P coatings do not lead to the graphitization of fibres during the high temperature sintering, i.e., the tensile properties of CFs are not influenced by the Ni-Co-P coatings. However, the interface boundary between coatings and Cu matrix (Fig. 6c) is not obvious because of the interface reaction. The metallurgical bonding interface can ensure the effective transfer of load from Cu matrix to fibres.

2.4 Effects of CFs content and different coatings on tensile properties of composites

The ultimate tensile strength (UTS), yield strength (YS), and elongation (EL) of CF/Cu(Ni-Co-P) composites with 10vol%, 20vol%, 30vol%, and 40vol% CFs fabricated by the vacuum hot pressing at 850 °C and 30 MPa for 40 min are shown in Fig.7. With increasing the CFs content, UTS and YS are firstly increased and then decreased. When CFs content is 30vol%, the CF/Cu(Ni-Co-P) composites have the highest UTS of ~415 MPa and YS of ~275 MPa, which are increased by 157.8% and 212.5%, compared with those of the copper matrix (UTS=161 MPa, YS=88 MPa), respectively. In addition, UTS and YS of CF/Cu(Ni-Co-P) composites with 40vol% CFs are 360 and 230 MPa, respectively, which are lower than those of the CF/Cu(Ni-Co-P) composites with 30vol% CFs.

For the composites reinforced by continuous fibres, the tensile properties are determined by the fibre strength and the fibre content, which can be predicted based on the law of mixtures, as expressed by Eq.(4)[26], as follows:

$$\sigma_c=\sigma_f V_f+E_m \varepsilon_f V_m \tag{4}$$

where σ_c and σ_f are UTS of the composites and fibres, respectively; V_f and V_m refer to the volume fraction of fibres and matrix, respectively; E_m and ε_f represent the elastic modulus of matrix and the fracture strain of fibres, respectively. Therefore, the tensile properties of the composites are increased with increasing the CFs content. However, when CFs content exceeds 30vol%, Eq.(4) is no longer suitable for the prediction of tensile properties of CF/Cu(Ni-Co-P) composites. The cavity defects in CF/Cu(Ni-Co-P) composites with 40vol% CFs may act as the starting points for the crack initiation and propagation, thereby leading to the fibre debonding and pullout, which reduces the load transfer efficiency of Cu matrix. Additionally, the measured tensile strength of the composite is far lower than the theoretical strength. There are two main reasons for this result. Firstly, the residual strength of CFs is decreased because of the partial diffusion between Ni and fibres during the hot pressing sintering[9], so it is less than their room-temperature strength. Secondly, the inhomogeneous loading capacity caused by the thermal expansion mismatch between fibres and matrix results in the failure of fibres, i.e., CFs cannot bear the load at the same time, therefore reducing the material strength.

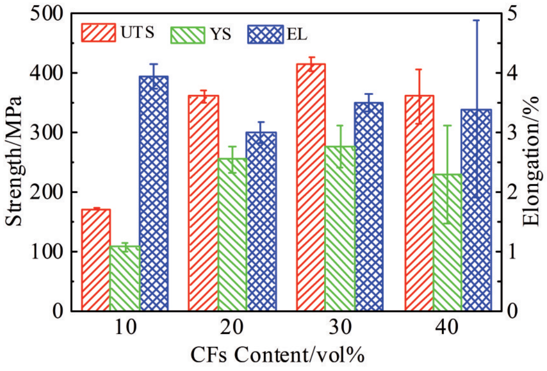

Fig.7 Tensile properties of CF/Cu(Ni-Co-P) composites with different CFs contents

Fig. 8 shows the tensile properties of Cu, CF/Cu, CF/Cu(Ni), and CF/Cu(Ni-Co-P) composites to investigate the effect of Ni-Co-P coatings on composites. It can be seen that YS and UTS of pure Cu are 88 and 160 MPa, respectively, which are much lower than those of other composites, although its EL is the highest among these composites. After the reinforcement with CFs, the tensile properties of CF/Cu (Ni), and CF/Cu(Ni-Co-P) composites are higher than those of the CF/Cu composite. Compared with those of CF/Cu composite, UTS (415 MPa) and YS (275 MPa) of CF/Cu(Ni-Co-P) composite are increased by 53.7% and 50.5%, respectively; while UTS (303 MPa) and YS (240 MPa) of CF/Cu(Ni) composite are increased by 12.2% and 30.4%, respectively. This result demonstrates that the alloy coatings have a better interface modification effect on the composite properties than the single-element metallic coatings do.

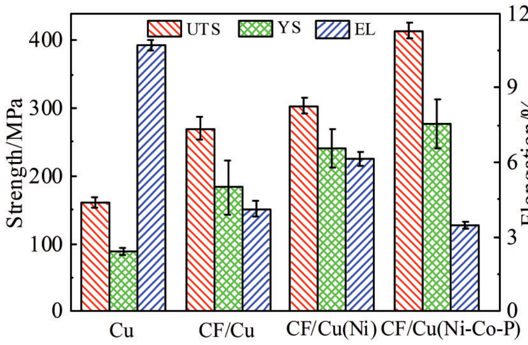

Fig.8 Tensile properties of Cu, CF/Cu, CF/Cu(Ni), and CF/Cu(Ni-Co-P) composites

2.5 Fracture mechanism of Cu matrix composites

The stress-strain curves of Cu, CF/Cu(Ni-Co-P), CF/Cu(Ni), and CF/Cu composites are shown in Fig.9a. According to the stress-strain curve of pure Cu, there is no obvious yielding point, and the wide plastic deformation zone indicates that the pure Cu possesses excellent plasticity and toughness. After the reinforcement with different CFs, the plastic deformation zones of composites have different degrees of shortening, which suggests that the failure modes of Cu matrix composites change and are determined by the interface bonding states based on the coating status. From the stress-strain curve of CF/Cu composite, the composites show linear elastic deformation when the stress is below 40 MPa. When the stress increases to ~40 MPa, the microcracks begin to initiate at the interface and are propagated to the Cu matrix, leading to the variation of deformation mode from linear elastic deformation to nonlinear plastic deformation. According to the fracture morphology of CF/Cu composite (Fig. 9b), the composite damage mainly includes three types: interface debonding, fibres fracture, and sliding. Thus, the failure mode is an accumulative fracture. For the CF/Cu(Ni) composite, the stress-strain curve has a similar tendency with that of CF/Cu composite, suggesting that the essential failure mode of composites does not change. According to the fracture morphology of CF/Cu(Ni) composite (Fig. 9c), the fibre pull-out length is shorter than that in the CF/Cu composite, which indirectly proves that the Ni coating can enhance the interface bonding strength and restrain the microcrack initiation and propagation, therefore significantly improving the tensile strength of composites and the critical stress of deformation. However, there are obvious interfacial debonding between the fibres and matrix, inferring that the failure mode of CF/Cu(Ni) composite is still the accumulative fracture. The elastic modulus of CF/Cu(Ni-Co-P) composite is similar to that of CF/Cu and CF/Cu(Ni) composites in the initial loading stage. With increasing the stress to ~275 MPa, the loading exceeds the UTS of partial fibres, so the deformation of composites changes from linearity to nonlinearity caused by the initiation and propagation of microcracks on fibres. The fibre debonding is restrained by the strong interface bonding strength between CFs and Cu matrix. With further increasing the loading, the fracture of partial fibres decreases not only the strength of fibres, but also the strength of matrix due to the crack propagation. These phenomena can decrease the elastic-plastic modulus with increasing the strain. As shown in Fig. 9d, the majority of the fibres and matrix break on the same plane at the same time, and only a few

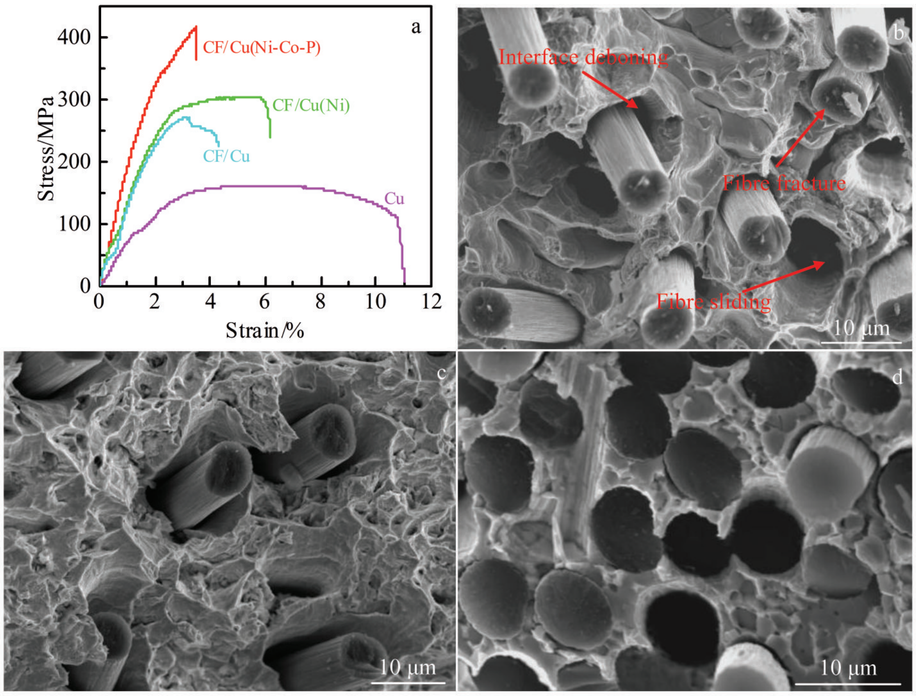

Fig.9 Stress-strain curves of pure Cu and Cu matrix composites (a); fracture morphologies of CF/Cu (b), CF/Cu(Ni) (c), and CF/Cu(Ni-Co-P) (d) composites

fibres are slightly pulled out. Thus, the failure mode of CF/Cu(Ni-Co-P) composite is the non-accumulation fracture.

3 Conclusions

1) Smooth and continuous Ni-Co-P coatings containing 61.4wt% Ni, 9.7wt% P, and 28.9wt% Co with the thickness of ~0.7 μm can be obtained on carbon fibres (CFs) by palladium-free electroless plating at 70 °C for 10 min.

2) The copper matrix composites reinforced by Ni-Co-P coated CFs, namely CF/Cu(Ni-Co-P) composites, with CFs reinforcement content of 10vol%, 20vol%, 30vol%, and 40vol% can be fabricated by the vacuum hot pressing sintering at 850 °C for 40 min. The tensile properties of CF/Cu(Ni-Co-P) composites are increased firstly and then decreased with increasing the CFs content. The CF/Cu(Ni-Co-P) composites with 30vol% CFs have the highest ultimate tensile strength of ~415 MPa and yield strength of ~275 MPa, while their elongation is inferior.

3) The failure mode of CF/Cu(Ni-Co-P) composites is the non-accumulation fracture, whereas that of other Cu matrix composites reinforced by CFs is the accumulation fracture.

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Ni-Co-P多元合金镀覆碳纤维增强Cu基复合材料的性能及断裂机制

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摘　要：采用无钯化学沉积法在碳纤维（CF）表面沉积厚度约0.7 μm、成分可控的Ni-Co-P合金镀覆层，并通过热压烧结工艺在850 ℃、30 MPa真空条件下分别制备纤维增强体体积分数为10%、20%、30%和40%的Ni-Co-P镀覆CF增强铜基复合材料CF/Cu(Ni-Co-P)、30%未修饰CF增强铜基复合材料CF/Cu和30% Ni镀覆CF增强铜基复合材料CF/Cu(Ni)。通过能谱仪、扫描电镜对CF/Cu(Ni-Co-P)中增强体及界面相元素分布、断口形貌进行观察，并采用电子万能试验机对复合材料拉伸性能进行测试。结果表明：在70 ℃水浴条件下沉积10 min可在CF表面获得厚度均匀、表面平整的Ni-Co-P多元合金镀覆层。CF/Cu(Ni)复合材料拉伸性能随着纤维含量的升高呈先提升后降低的趋势，30% CF/Cu(Ni-Co-P)复合材料的抗拉伸强度和屈服强度最高。在增强体含量相同条件下（30%），CF/Cu(Ni-Co-P)复合材料的力学性能明显优于CF/Cu(Ni)和CF/Cu复合材料，断裂机制为非积聚型失效。

关键词：Ni-Co-P修饰碳纤维；铜基复合材料；拉伸性能；断裂机制

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