Rare Metal Materials and Engineering
Volume 50, Issue 1, January 2021
Available online at www.rmme.ac.cn

Science Press

Cite this article as: Rare Metal Materials and Engineering, 2021, 50(1): 0085-0094.

ARTICLE

Property of Cu-Graphene Composite Coatings on Quartz Fiber Surface

Song Qiliang[1,2], Hu Zhenfeng[1], Tu Long[1], Kong Lingchao[1], Liang Xiubing[1], Liu Erbao[2]

[1] National Innovation Institute of Defense Technology, AMS, Beijing 100071, China; [2] Institute of Corrosion Science and Surface Technology, School of Materials Science and Chemical Engineering, Harbin Engineering University, Harbin 150001, China

Abstract: In order to obtain the metal-coated fiber with high temperature resistance and long service life, Cu-based coating was plated on quartz fiber surface by electroless plating and studied by material characteristic parameters measurement. SEM, EDS, XPS and Raman spectra were used to characterize the microstructure of graphene sheet and Cu-based coating. Electrochemical workstation and nano indentation instrument were used to test the properties of metal coating. The bonding properties of Cu-based coating and fiber substrate were analyzed by thermal shock method. The results show that compared with Cu coating, Cu-graphene coating has more compact structure, better quality and fine grain, with the hardness and elastic modulus increased by 35.30% and 34.0% respectively. The corrosion potential E_{corr} of Cu-graphene coating is increased by 32.3%, and the corrosion current i_{corr} is decreased by 22.5%. The corrosion resistance of Cu-graphene coating improved. The surface of quartz fiber is electroless-coated with Cu metal coating, which can overcome the problem of burning out due to excessive local temperature of optical filter of optical fiber cladding in practical application. The coating has no blocking on signal transmission inside the optical fiber. Graphene can improve the forming quality of optical fiber surface coating, improve corrosion resistance and other properties, which is of great significance to improve the service life of optical fiber.

Key words: electroless plating; corrosion resistance; graphene; quartz optical fiber; Cu-graphene composite coating

With the rapid development of science and technology, material with more comprehensive properties is greatly needed on the demand of advanced technology[1-3]. As the supporting material of optical equipment, quartz fiber can be divided into fiber core, inner cladding, outer cladding and coating layer[4]. At present, the coating layer is mainly composed of organic matter, which has low melting point and corrosion resistance, restricting the application of optical fiber in extreme environments[5-10]. Therefore, the surface modification of optical fiber has gradually become a new research hotspot. Electroless metal coating on optical fiber surface is one of the effective methods to improve the overall properties of optical fiber[11-13]. Electroless plating technology has the advantages of high plating efficiency, good coating quality, and low cost[13,14]. It has an important application prospect in the field of material modification, equipment protection and remanufacturing. Electroless plating technology can effectively coat the non-metallic surface with metal, generating metallic characteristics[15-18]. Graphene not only has outstanding electrical and thermal conductivity, but also has strong corrosion resistance and hydrophobic properties, known as the "king of new materials"[19-22]. The hardness, thermal conductivity and corrosion resistance of the coating can be effectively

Received date: January 05, 2020
Foundation item: National Natural Science Foundation of China (51605491, 51505484)
Corresponding author: Hu Zhenfeng, Ph. D., Associate Researcher, National Innovation Institute of Defense Technology, AMS, Beijing 100071, P. R. China, E-mail: hu_zhenfeng@sina.com

improved by compounding graphene into the metal coating[23,24].

In this study, Cu coating was attached on the surface of quartz fiber by electroless coating technology. In addition, the Cu-graphene composite coating was prepared by compounding the graphene into the metal coating. The microstructure of Cu coating and Cu-graphene composite coating gluing on optical fiber was compared and analyzed. At the same time, light transmission experiment of optical fiber with different coating was conducted.

1 Experiment

1.1 Experimental setup and devices

Two kinds of optical fibers, model 20/400 (fiber core diameter 20 μm and fiber cladding diameter 400 μm) and model 220/242, were selected as the matrix materials. Two kinds of optical fibers were intercepted to 20 cm and put into alcohol for ultrasonic cleaning for 10 min, then taken out for following process. The graphene lamellar material was prepared by REDOX method, and the single lamellar structure accounted for more than 96%. Fig.1 shows the SEM morphology of graphene lamella. It can be clearly demonstrated that the graphene sheet layer has a higher degree of transparency and folds due to thermal instability during preparation.

The main experimental devices include SEM, magnetic stirrer, pen type pH meter, analytical balance, water bath pot, ultrasonic cleaning instrument, XPS, Raman spectrometer, electrochemical workstation

1.2 Chemical plating formula and process

The main components of the chemical plating solution is shown in Table 1. Attention should be paid to the sequence of chemical reagent when preparing electroless copper plating solution. First, 500 g $C_4H_4KNaO_6{\cdot}4H_2O$, 150 g $CuSO_4{\cdot}5H_2O$, 20 g $NiSO_4{\cdot}6H_2O$, 250 mL HCHO, an appropriate amount of stabilizer and 20 g Na_2CO_3 were dissolved in a small amount of deionized water. Next, they were poured into the beaker in the same order as above, stirred, and then NaOH solution was added to adjust pH to 12.6. Finally, the volume of deionized water was increased to 1 L, and the water was filtered for use.

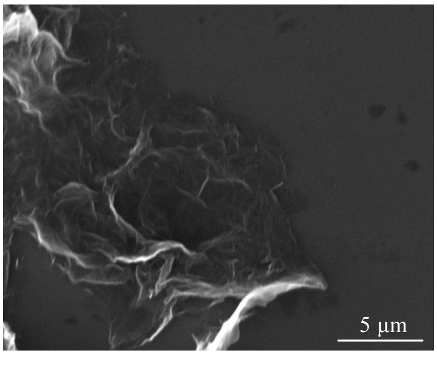


Fig.1 SEM image of graphene lamella

However, in the preparation of composite plating solution, graphene dispersion should be added to Cu plating solution before constant volume to ensure that pH value of the plating solution keeps constant. After adding the graphene lamellae, the solution was strongly stirred for at least 2 h under ultrasonic condition until the color of the plating solution is uniform, so as to ensure that the graphene is evenly dispersed into the plating solution.

In order to ensure the integrity of optical fiber materials, this experiment adopted the pretreatment process without coarsening steps to prepare bare optical fiber (Fig.2). The main four steps of electroless plating include removal of coating layer, sensitization, activation and electroless plating.

2 Results and Discussion

2.1 Microstructure

The coating was characterized by Raman spectrum with 514 nm laser source, as shown in Fig.3. Fig.3a shows the composite coating Raman test selection region. In order to visually and clearly observe the graphene, thin nitric acid was used to slightly etch the coating surface between 5 and 10 s before Raman test. As shown in Fig.3a, the black lamellar material is embedded on the coating surface. Fig.3b is the Raman spectrum of the coating. It can be seen that the characteristic peak D and characteristic peak G of graphene materials appear near 1340 and 1580 cm^{-1}, respectively, and the characteristic peak 2D of severe deformation occurs within the range of 2500~3000 cm^{-1}. The main reason is that the graphene in the composite coating is coated with Cu, and the coated metal win cause 2D peak blue shift (right shift). Secondly, the agglomeration

Table 1 Main components of chemical plating solution

Substance	Concentration
$C_4H_4KNaO_6{\cdot}4H_2O$	500 g/L
$CuSO_4{\cdot}5H_2O$	150 g/L
$NiSO_4{\cdot}6H_2O$	20 g/L
HCHO	250 mL/L
Na_2CO_3	20 g/L

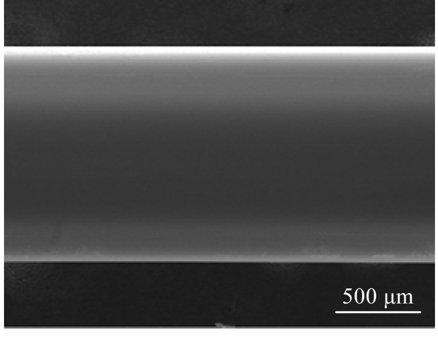


Fig.2 SEM image of bare optical fiber

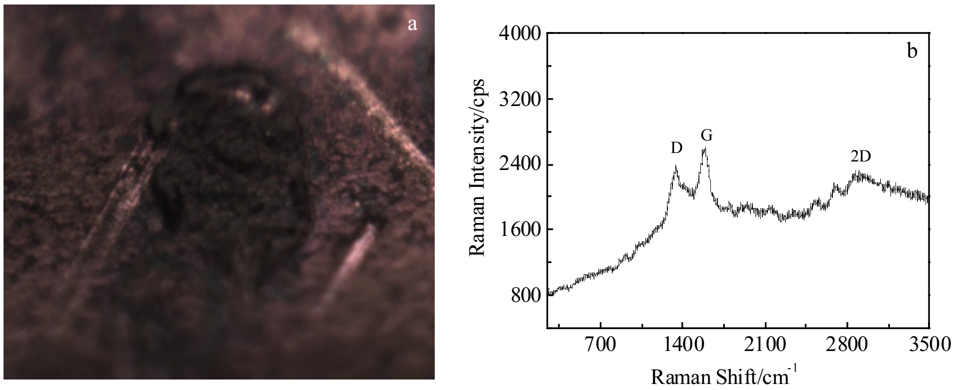


Fig.3 Raman test for Cu-graphene coating: (a) composite coating Raman test selection and (b) Raman spectrum

of graphene causes the lamination to be superimposed. When the laser passes through the stacked layers of graphene, it will the half height and full width of 2D peak, and peak wavelength will be blue shifted.

Then SEM was used to characterize the metal-coated fiber and the results are shown in Fig.4. The micromorphology of Cu-based coatings at different multiples is demonstrated. Comparing Fig.4a and 4d, 4c and 4f, we can come to the conclusion that the admixture of graphene into the coating can significantly improve the quality of the coating with the surface being denser and the coating becomes shinier. Among them, the pure Cu coating grain is relatively coarse, while the Cu-graphene composite coating grain is obviously refined, showing needle shape and compact arrangement. Fig.4b and 4c are SEM images of Cu coating under high multiple, and it can be seen that the grain distribution of the coating is uniform. Fig.4e and 4f shows the composite coating with the addition of graphene materials. The coating has more luster, uniform distribution and good sensitization, compared with Cu coating. This is because the proper amount of graphene sheet layer and the precipitated metal in the process of plating and deposition are constantly filled into the surface defects of the original coating, thus changing the overall compactness of the composite coating. Fig.4f are SEM images of high-power composite coating. It can be seen that the addition of graphene material can lead to prominent “particles” on the surface of the coating to different degrees, which is caused by graphene sheet. In the process of electroless plating, an appropriate amount of graphene sheet can be adsorbed to the surface of the substrate plate, providing nucleated particles for the metal coating. At the same time, its excellent conductive and thermal properties will also accelerate the movement of ions in the plating solution, making metal ions in the plating solution to be preferentially reduced on the surface of graphene. At the same time, the nucleation and growth of grains are rapid, which leads to the surface of graphene being coated with metal particles, forming raised “particles”. According to the Raman spectrum analysis above, the graphene sheet is uniformly distributed in the Cu coating as the second phase, accelerating the deposition of metal ions.

2.2 Cross section morphology

In order to analyze the relevant properties of the coating more accurately, the section of the coating was characterized and analyzed by SEM. Fig.5 shows the section microstructure of the copper base coating. As shown in Fig.5a, the pure Cu coating is closely bound to the matrix, with no obvious gap, and the coating thickness is 5~10 μm. Fig.5b shows Cu-graphene coating containing 0.05 g/L graphene. Compared with Fig.5a, the coating is more closely bound to the substrate, the plating thickness is slightly increased, and there are no internal pores and penetration cracks in the coating. The introduction of graphene sheet layer significantly improves the quality of Cu coating.

2.3 Coating XPS analysis

Cu-based coatings were tested by X-ray photoelectron spectroscopy. The test results are shown in Fig.6 and Fig.7, and the existence forms of different feature elements are summarized, as shown in Table 2. As can be seen from Fig. 6, Cu elements exist in elemental form in Cu coating. Fig. 7a and Fig.7b show that Cu elements in Cu-graphene coating exist in elemental form. C element is mostly in elemental form, and a small amount in the form of $C_6H_5CH_3$, CH_3CONH_2 and CCl_4.

Table 2 Existing forms of elements in coating

Coating type	Cu	C
Cu	Elemental	-
Cu-graphene	Elemental	Elemental/$C_6H_5CH_3$/ CH_3CONH_2/CCl_4

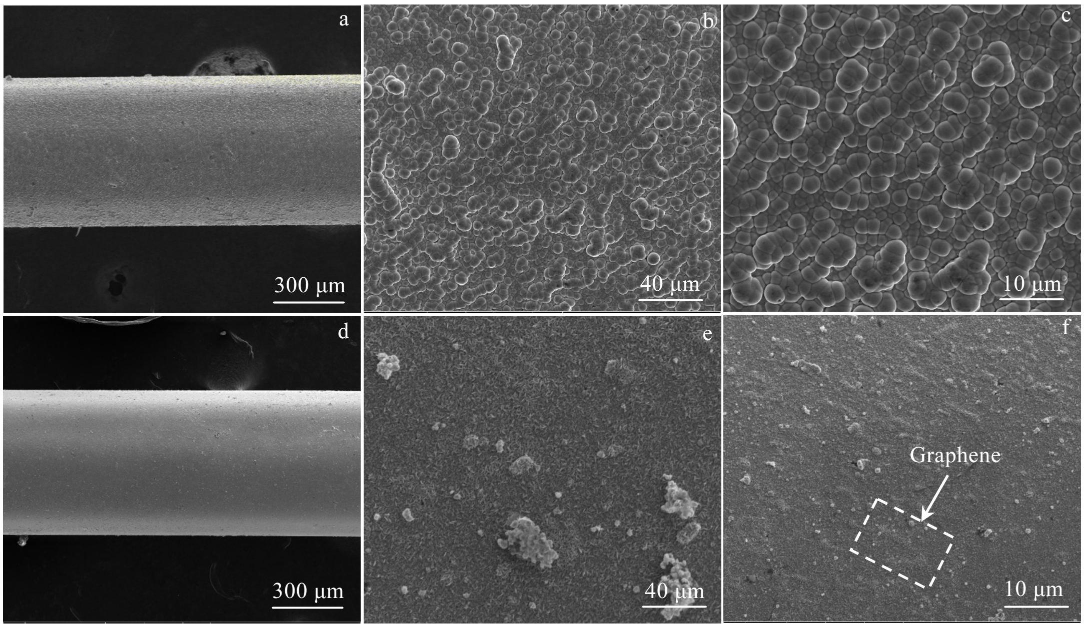


Fig.4 Optical fiber microtopographies of different coatings: (a~c) Cu coating and (d~f) Cu-graphene coating

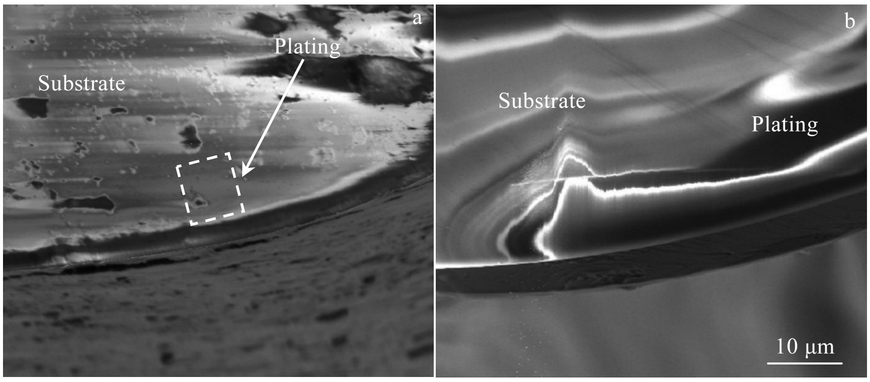


Fig.5 SEM morphologies of fiber optic section of metal coating: (a) Cu coating and (b) Cu-graphene coating

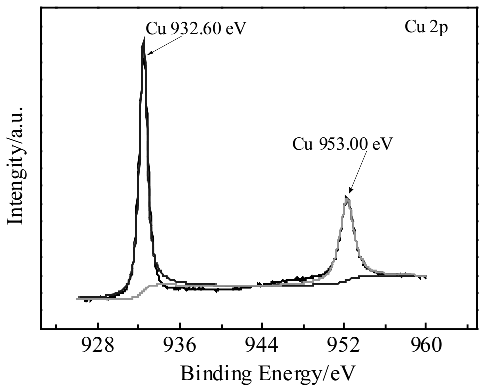


Fig.6 XPS deduplication of characteristic elements of Cu coating

2.4 Coating hardness and modulus of elasticity

Table 3 shows the average hardness of the coating. Because the pattern of the metallic coating fiber is small, it can not be characterized by conventional durometer, so this experiment adopts nano indentation method to test the sample with minimal damage. According to Table 3, the average hardness of Cu coating is 4.50075 GPa and the average hardness of Cu-graphene coating is 6.0885 GPa. With the adding of graphene ingredients, the hardness of Cu-based coating is improved significantly by 35.3%. The reason for this phenomenon is that the graphene sheet can be dispersed and distributed in the coating, preventing the dislocation movement of the metal and playing the role of dispersion strengthening. The monolayer itself has high strength. The graphene sheet adsorbed on the surface

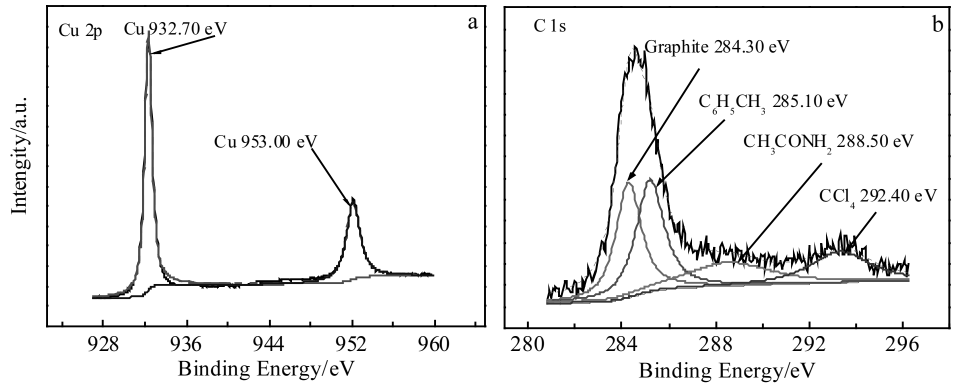


Fig.7 XPS deduplication of Cu-graphene coating characteristic elements: (a) Cu element and (b) C element

Table 3 Average hardness and elastic modulus *E* of optical fibers with different metal coatings

Coating type	Cu	Cu-graphene
Hardness/GPa	4.50075	6.0885
E/GPa	55.188	73.954

of the coating provides more nucleation particles for the rapid deposition of metal ions, which can greatly increase the number of grains, refine the grains and play the role of fine grain strengthening.

Fig.8 shows the load-displacement curve of the coating. It can be concluded that when the graphene ingredient is introduced into the plating solution, the elastic modulus *E* of Cu-graphene coating is significantly improved. According to Table 3, the elastic modulus of Cu coating is 55.188 GPa, and that of Cu-Graphene coating is 73.954 GPa, which is 34.0% higher than that of pure Cu coating. This is because the graphene material itself has a large elastic modulus, up to 1 TPa. But the original structure is not damaged after the introduction of composite coating. Meanwhile, when the content of graphene in the plating solution is appropriate, its even dispersion in the coating enhances the high elastic modulus characteristics.

2.5 Corrosion resistance analysis of coating

Table 4 shows the self-corrosion potential and self-corrosion current density of the coating in 3.5wt% NaCl solution. Fig.9 shows the polarization curves of different coatings in 3.5wt% NaCl solution. According to Table 4 and Fig.9, the corrosion potential E_{corr} of Cu-graphene coating moves forward to -0.176 mV and that of pure Cu coating is –0.25954 mV, improved by 32.3%. Cu-Ge coating corrosion current density i_{corr} negatively shifts to –8.17758 μA·cm^{-2} and that of pure Cu plating is –6.33538 μA·cm^{-2}, decreased by 22.5%. Cu-graphene shows significant passivation zone. According to the distribution of self-corrosion potential

Table 4 Self-corrosion potential E_{corr} and self-corrosion current density i_{corr} of coating in 3.5wt% NaCl solution

Coating type	Cu	Cu-graphene
E_{corr}/V	-0.25954	-0.176
i_{corr}/μA·cm^{-2}	-6.33538	-8.17758

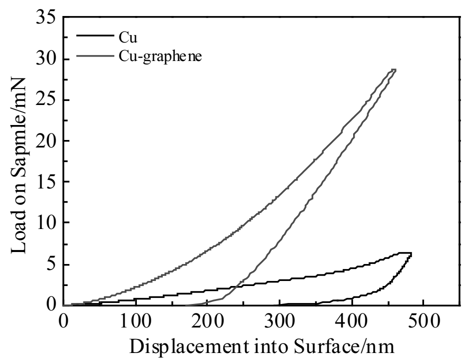


Fig.8 Load-displacement curves of Cu coating

and self-corrosion current, it can be determined that the corrosion resistance of Cu-Ge coating is higher than that of Cu coating. The reasons for this phenomenon are as follows. First of all, according to Fig.4, the surface of the Cu coating is relatively rough, with large grains and obvious gaps. However, Cu-Ge coating has a flat surface, higher sensitization, no grain gap and no corrosion medium can enter the coating structure, which slows down the electrochemical reaction process and improves the corrosion resistance of the composite coating. Secondly, graphene has a high conductivity, which can hinder the electron transfer during the corrosion process and slow down the corrosion. Finally, due to the high hydrophobicity of graphene materials, appropriate amount of graphene can be uniformly and closely deposited in the coating after being mixed into the plating solution, forming a “maze effect” and protecting the coating substrate against corrosion. The path of the corrosion medium to the substrate is greatly extended, and the corrosion resistance of the composite coating is improved.

Fig.10 is the SEM image of the electrochemical corrosion microstructure of coatings. As shown from Fig.10a, the coating surface does not fall off and is pitted. In Fig.10b, it can be clearly seen that after electrochemical corrosion, the bright white strip or granular graphene flake layer is embedded on the surface of the coating, and the coating has a good integrity and is not damaged. Compared with Fig.10a, the degree of corrosion damage of the coating is significantly reduced.

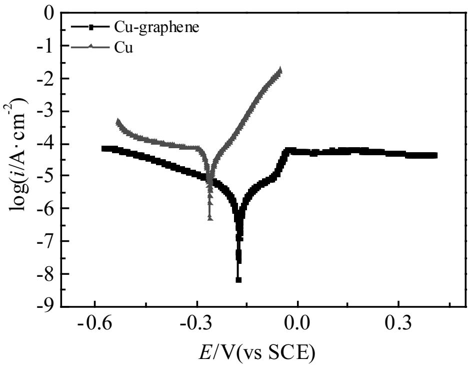


Fig.9 Polarization curve of optical fiber for different metal coatings

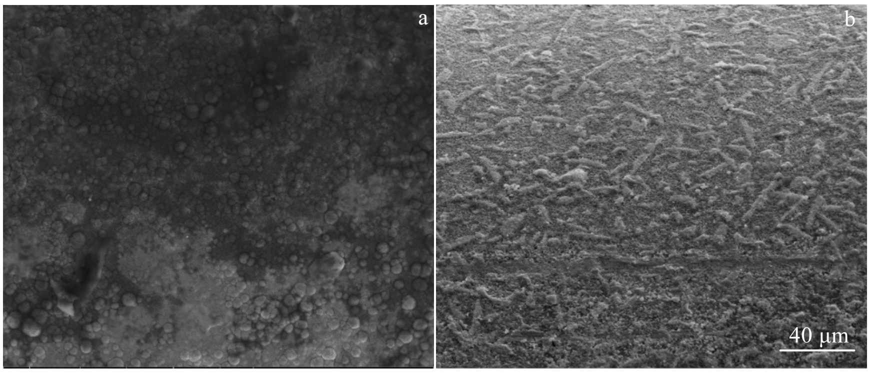
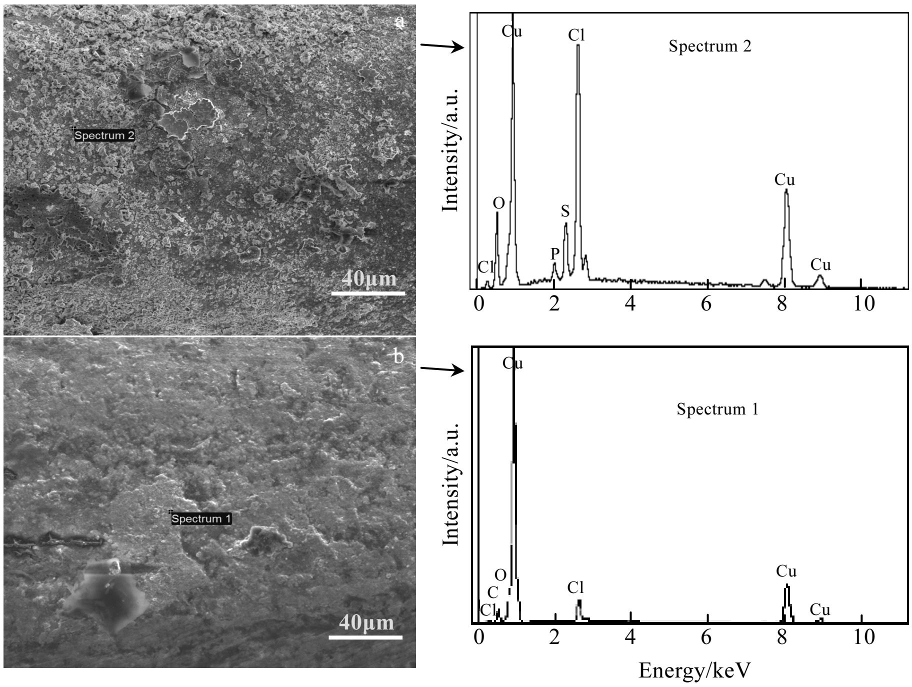


Fig.10 SEM images of fiber corrosion of different metal coatings: (a) Cu coated optical fiber and (b) Cu-graphene coated optical fiber

Fig.11 shows the EDS analysis results of electrochemical corrosion areas of different coatings. Combined with Fig.9 and Fig.10, it can come to the conclusion that corrosion products of different contents appear on the coating surface after electrochemical corrosion. The main elements in the corrosion products of Cu coating are Cu, Ni, P, O, S and Cl. The main elements of corrosion products in Cu-graphene coating are Cu, C, O, Cl. Additionally, the Cu content of the coating substrate material in Fig.11a is 45.72wt%. However, in Fig.11b, Cu content is 82.67wt%, and O and Cl corrosion products contain less elements. In the composite coating, the graphene sheet structure can be well interspersed between multiple metal grains, reducing the grain size and filling the gap of the coating, and thus slowing down the corrosion process of the coating.

2.6 Light test

According to the conducting light test on the electroless plated optical fiber, the results are shown in Table 5 and Fig. 12. Compared with No.2 and No.3, it can get conclusion that with the increasement of plating time, the thicker the coating thickness, the better the performance of the heat pipe. Under the same electroless plating time, when the surface temperature of the metal fiber reaches 100 ºC, the input power of the Cu coating is 6.73 W, while the performance of the Cu-graphene coating heat pipe does not change significantly. According to Fig.12, when the temperature of a certain point on the surface of the fiber reaches 100 ºC, the point is mostly the junction between the surface metal coating and the uncoated metal. The average temperature of the metal coating on the surface is 23.9 ºC, which illustrates that the surface of the optical fiber containing metal coating can continuously increase the laser power. The reasons for the above phenomenon can be summarized as the following two points. First, it can be concluded from the properties of the material that the thermal conductivity of pure copper metal is 401 W/mK, and the metallic copper has higher thermal conductivity than the organic coating layer, so the heat pipe performance



Element	wt%	at%
O	18.07	39.03
P	1.66	1.85
S	5.62	6.06
Cl	28.93	28.20
Cu	45.72	24.86
Total	100.00	100.00

Element	wt%	at%
C	6.72	23.99
O	5.03	13.49
Cl	5.58	6.75
Cu	82.67	55.78
Total	100.00	100.00

Fig.11 EDS results of corrosion area of optical fiber for different metal coatings: (a) Cu coated optical fiber and (b) Cu-graphene coated optical fiber

of Cu coating fiber is better. Second, the addition of graphene does not significantly improve the thermal management performance of the coating material, because the coating material on the surface of the quartz fiber is too thin, only 10~15 μm, and the graphene sheet is distributed in different shapes and positions in the coating. According to relevant literatures, graphene laminates have the best thermal conductivity in the plane spreading direction, while the thermal conductivity in the vertical plane direction is average. Therefore, it can be concluded statistically that the content of graphene laminates perpendicular to the axial part of the optic fiber composite coating is relatively small, which means that graphene has little influence on the thermal management performance of the composite coating.

To sum up, quartz fiber can overcome the burning loss and other problems in practical application by electroless plating Cu metal coating on its surface, and continue to increase the laser power on the basis of experiments. Although the mixture of graphene has no obvious effect on the thermal conductivity of the coating, it can not only improve the quality of the coating on the optical fiber surface, but also improve the anti-corrosion and tensile properties. Generally speaking, graphene is of great significance in improving the service life of optical fiber.

Table 6 and Fig.13 are the test results of optical fiber sensing performance of the model 220/242 metal coating. For further study of the influence of chemical gold-plating on optical fiber signal sensing, Cu coating and Cu-graphene coating were applied in the same process on the surface of model 220/242 optical fiber, and the optical transmittance of metallic coating fiber was tested. As can be seen from Table 6, the light transmission power of model 242 optical fiber signal is above 97.0%, and its signal light power loss is small, which meets the requirements of 220/242 metal-coated optical fiber in work.

Table 5 Metal-coated fiber pump light test

No.	Coating types	Current/A	Power input (100 ºC)/W	Through the power/W(%)	Time/h
1	-	14.00	69.50	69.00 (99.3)	-
2	Cu	1.08	2.10	1.34 (63.6)	0.5
3	Cu	1.85	6.73	5.66 (84.1)	1.0
4	Cu-Ge	1.50	6.64	5.58 (84.1)	1.0

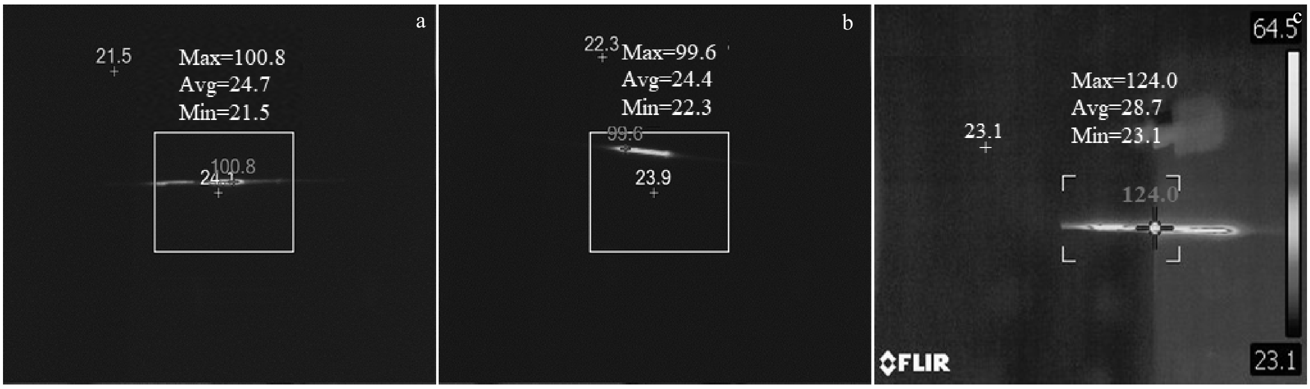


Fig.12 Infrared thermal sensation of optical fiber with different metal coatings: (a) Cu coating for 0.5 h, (b) Cu coating for 1.0 h, and (c) Cu-graphene coating for 1 h

Table 6 Optical signal sensing test for different metal coatings

No.	Coating types	Current/A	Power input (100 ℃)/W	Through the power/W(%)
1	Cu	2.14	30.3	9.74 (97.4)
2	Cu-Ge	2.14	31.9	9.82 (98.2)

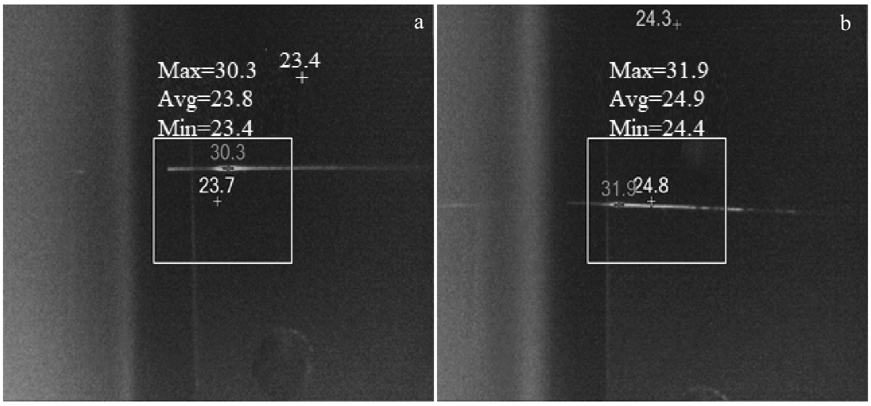


Fig.13 Infrared thermal images of model 220/242 metal coating optical fiber: (a) Cu coating and (b) Cu-graphene coating

It can be seen from Fig.13 that there is no obvious temperature rise on the surface of the metal coating during signal light transmission. When the input power is 10 W, the average temperature of the fiber surface coating is 30 ºC, and the temperature distribution field is uniform, because the signal light and pump light transmission path is different. When the signal light is injected into the fiber, the signal light mainly propagates along the fiber core and reflects at the interface between the fiber core and the inner cladding. When pumping light is injected, it is transmitted along the inner cladding layer and reflects at the interface between the inner cladding layer and the outer cladding layer. In this experiment, the model 20/400 metal-coated fiber may destroy outsourcing layer of the fiber when the surface coating layer is removed in the pre-treatment, so that the pump light can directly contact the metal coating and generate heat. In the model 220/242 fiber test, there is no signal light exposure and no large amounts of heat generation.

Fig.14 is optical fiber spectrogram of model 220/242 metal coating. The influence of metal coating on optical fiber transmission was analyzed by measuring the change of signal wavelength. The central wavelength of laser refers to the wavelength corresponding to the central position of the half-height and full-width of the spectrum measured at a certain temperature and rated power. The half-height and full-width of the laser refers to the wavelength difference when the intensity on both sides of the peak value of the spectrum drops to half of the peak value[25]. As shown in Fig. 14, each coating spectrum has a narrow and sharp peak value since the width of peak is narrow, and the central wavelength is approximately the peak value. Among them,

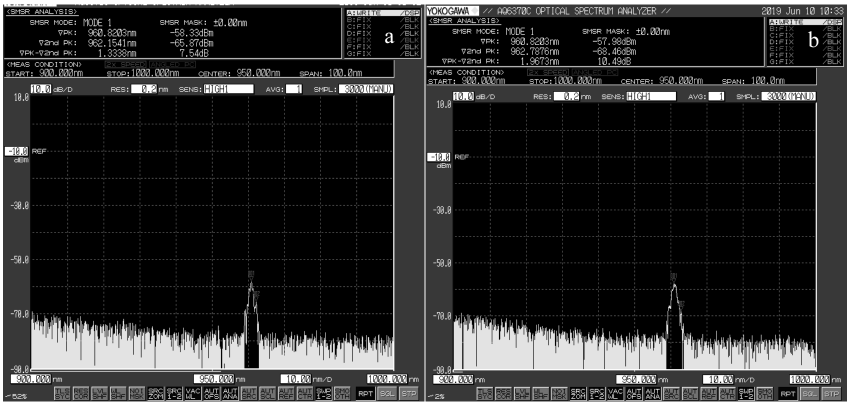


Fig.14 Optical fiber spectrogram of model 220/242 metallic coating: (a) Cu coating and (b) Cu-graphene coating

the central wavelength of Cu coating fiber is 960.8203 nm, and that of Cu-graphene coating fiber is 960.8203 nm, with no offset. It can be seen that the chemical gold-plated coating on the surface of quartz fiber has no effect on the signal and light transmission of the fiber, which meets the application conditions of the metal-coated fiber in information sensing.

3 Conclusions

1) Compared with Cu coating, Cu-graphene coating prepared by electroless plating technology has denser microstructure, smaller grains and more luster, and no cracks or other defects exist, showing better quality. In Cu coating and Cu-graphene coating, Cu element exists in elemental form, C element exists mainly in elemental form with only a small amount in the form of $C_6H_5CH_3$, CH_3CONH_2 and CCl_4.

2) The average hardness and elastic modulus of metallic Cu coating are 4.50075 and 55.188 GPa, respectively, while the average hardness and elastic modulus of Cu-graphene coating are 6.0885 GPa and 73.954 GPa, respectively. Compared with Cu coating, the mixture of graphene material significantly improves the quality of Cu-graphene coating, with hardness and elastic modulus increased by 35.3% and 34.0%, respectively.

3) Through electrochemical corrosion, the corrosion potential E_{corr} of Cu-graphene coating is increased by 32.3% and the corrosion current i_{corr} is decreased by 22.5% compared with Cu coating. Moreover, there is an obvious passivation zone in the polarization curve of Cu-graphene coating.

4) According to the pump light test, the surface of the quartz fiber is electroless-coated with Cu metal coating, which can overcome the problem of burning out due to high local temperature of the optical filter of the fiber cladding in practical application. The introduction of graphene sheet layer has no obvious influence on the thermal conductivity of the coating, but it can improve the quality, anticorrosion and tensile resistance of the surface coating, which is of great significance for improving the service life of optical fiber. The chemical gold-plated coating on the surface of quartz fiber has no effect on the signal light transmission in fiber, which meets the application conditions of metal-coated fiber in information sensing.

References

1 Liu Deming, Sun Junqiang. *Fiber Optics*[M]. Beijing: Science and Technology Press, 2008 (in Chinese)
2 Feng Hao, Qing Chun, Han Bing *et al*. *Mechanical and Electronic Components*[J], 2018, 38(4): 50
3 Zhang Lisong. *Thesis for Master Degree*[D]. Xi'an: Xi'an Petroleum University, 2018 (in Chinese)
4 Chen Jijing. *Innovation and Application of Science and Technology*[J], 2018, 29: 66
5 Song Bin, You Qingliang, Wang Yazhen. *Journal of Jianghan University*[J], 2015, 43(2): 127 (in Chinese)
6 Guo Yongjie, Zhao Songqing, Dou Haixiao *et al*. *Aviation Weapon*[J], 2017(6): 59 (in Chinese)
7 Zhou Wenxiu, Hou Wenbo, Zhang Haijun. *Chinese Medical Device*[J], 2018, 42(5): 354 (in Chinese)
8 Zhu Yuehong, Wen Jihua. *Surface Technology*[J], 2018, 47(7): 241
9 Lu Mingyang. *Thesis for Master Degree*[D]. Nanchang: Nanchang University, 2014 (in Chinese)
10 Yao Wenli, Guan Congsheng, Wang Jing *et al. Photoelectric Technology Application*[J], 2012, 27(6): 46, 54
11 Wang Yubo. *Thesis for Master Degree*[D]. Nanchang: Nanchang University, 2017 (in Chinese)
12 Yang Ke, Li Yulong, Li Xuewen. *Laser and Infrared*[J], 2018, 48(6): 769
13 Wang Hao, Feng Zhenglong. *Chemical Technology and Development*[J], 2018, 47(8): 37
14 You Lian. *Thesis for Master Degree*[D]. Wuhan: Wuhan University of Technology, 2010 (in Chinese)

15 Gao Hongjiang. *Thesis for Master Degree*[D]. Qingdao: Qingdao University of Science and Technology, 2016 (in Chinese)
16 Fang Meng, Hu Ling, Shi Changdong *et al*. *Transactions of Nonferrous Metals Society of China*[J], 2016(3): 1
17 Jiang Jinjin, Wu Wangping, Yuan Tongxin *et al*. *Electroplating and Finishing*[J], 2017, 39(10): 16
18 Xu Xukun, Zhao Dan, Wan Decheng *et al*. *Electroplating and Finishing*[J], 2016, 38(3): 27
19 Yang Chen, Liu Dingfu. *Electroplating and Environmental Protection*[J], 2015, 35(2): 17
20 Warner J H. Translated by Fu Lei. *Graphene: Basic and Emerging Applications*[M]. Beijing: Science Press, 2014 (in Chinese)
21 Iijima S. *Nature*[J], 1991, 354: 56
22 Zeng Fanyan. *Thesis for Master Degree*[D]. Changsha: Hunan University, 2013 (in Chinese)
23 Green A A, Hersam M C. *Phys Chem Lett*[J], 2010(1): 544
24 Liu Jianjian, Zhu Chengyi, Li Guangqiang *et al*. *Materials Protection*[J], 2018, 51(3): 77
25 Li Ke. *Thesis for Master Degree*[J]. Changsha: Hunan University, 2011 (in Chinese)

石英光纤表面 Cu-石墨烯复合镀层的性能研究

宋启良[1,2]，胡振峰[1]，涂　龙[1]，孔令超[1]，梁秀兵[1]，刘二宝[2]

(1. 军事科学院国防科技创新研究院，北京 100071)

(2. 哈尔滨工程大学 材料科学与化学工程学院 腐蚀科学与表面技术研究所，黑龙江 哈尔滨 150001)

摘　要：为制备耐高温、寿命长的金属镀层光纤，利用化学镀技术在石英光纤表面制备 Cu 基镀层。同时将石墨烯片层材料引入镀液，制备了 Cu-石墨烯复合镀层。采用扫描电镜（SEM）、能谱仪（EDS）、X 射线光电子能谱仪（XPS）、Raman 光谱仪等，对石墨烯片层和 Cu 基镀层的微观形貌进行表征。利用电化学工作站、纳米压痕仪等对金属镀层的性能进行测试。利用热震法对 Cu 基镀层与光纤基体的结合性能进行分析，同时对金属镀层光纤进行导光测试。结果发现：Cu-石墨烯镀层相对 Cu 镀层，镀层组织致密，晶粒细小，质量更优。Cu-石墨烯镀层硬度、弹性模量分别提升了 35.3%、34.0%。Cu-石墨烯镀层的腐蚀电位 E_{corr} 提升了 32.3%，腐蚀电流 i_{corr} 减小了 22.5%，其耐蚀性能明显提升。石英光纤表面化学镀覆 Cu 金属镀层，能够克服光纤包层光滤除器在实际应用中因局部温度过高而烧损等问题，同时对光纤的信号光传递并无影响。石墨烯片层对光纤对表面镀层质量、提高防腐等性能影响较大，在提升光纤使用寿命方面具有重要意义。

关键词：化学镀；耐腐蚀；石墨烯；石英光纤；Cu-石墨烯复合镀层

作者简介: 宋启良，男，1993 年生，硕士生，哈尔滨工程大学材料科学与化学工程学院腐蚀科学与表面技术研究所，黑龙江 哈尔滨 150001，E-mail: song_qiliang@163.com