Iraqi Journal of Physics, 2020 *Vol.18, No.47, PP. 55-61*

DOI: 10.30723/ijp.18.47.55-61

Enhancement of Thermal Stability and Wettability for Epoxy/Cu Coated Carbon Fiber Composites

Reem Y. Mahmood, Aseel A. Kareem

Department of Physics, College of Science, University of Baghdad, Baghdad, Iraq

Corresponding author: aseel.a@sc.uobaghdad.edu.iq

Abstract

This research study the effect of surface modification and copper (Cu) coated carbon fiber (CF) surface on thermal stability and wettability of carbon fiber/epoxy (EP) composites. The TGA results indicate that the temperature of 5% weight loss (T_5) for Ep/Cu coated CF composite increased to 322 ºC when compared with Ep/CF and Ep/chemical treated CF composite. The temperature of 10 wt.% (T_{10}) and the degradation temperature (T_d) of the Ep/CF composite are 266 and 522°C, respectively, with the chemical treatment and Cu coating, T_{10} and T_d of Ep/CF significantly increased to (310.3, 345 and 543, 565.5) ˚C. Ep/ CF composites showed lower thermal conductivity than that of Ep/Cu coated CF composite. After heat treatment, thermal stability and thermal conductivity of Ep/modified CF composite increased. The contact angle for CF decreased slightly after chemical treatment to 105º. After Cu electroless coating, the carbon fiber showed a contact angle of 82º. Heat treatment significantly improved the wettability of Cu coating CF showing 32º of contact angle at 250 ºC.

Key words

Reducing agent, carbon fiber modification, copper plating, complexing agent.

Article info.

Received: Sep. 2020
Accepted: Nov. 2020
Published: Dec. 2020

تعزيز الاستقرار الحراري وقابلية الترطيب لمتراكبات الإيبوكسي/ ألياف الكربون المطلية بالنحاس

ريم ياسر محمود, أسيل عبد الامير كريم

قسم الفيزياء, كلية العلوم, جامعة بغداد, بغداد, العراق

الخلاصة

يدرس هذا البحث تأثير تعديل السطح وطلاء سطح ألياف الكربون (CF) بالنحاس (Cu) على الأستقرار الحراري وقابلية الترطيب لمتراكبات ألياف الكاربون/ الإيبوكسي(Ep). تشير نتيجة التحليل الحراري الوزني (TGA) إلى زيادة نسبة فقدان الوزن بنسبة 5٪ (T_5) لمتراكبات الايبوكسي/ الياف الكاربون المطلية بالنحاس إلى 322 درجة مئوية عند مقارنتها بمتراكبات الايبوكسي/ الياف الكاربون ومتراكبات الايبوكسي/ الياف الكاربون المعالجة كيميائيا. نسبة 10٪ بالوزن (T_{10}) ودرجة حرارة التحلل (T_d) لمتراكبات الايبوكسي/ الياف الكاربون هي 266 و 522 درجة مئوية ، على التوالي، مع المعالجة الكيميائية والتغليف بالنحاس، تزداد T_{10} و T_d لمتراكبات الايبوكسي/ الياف الكاربون بشكل ملحوظ إلى (310.3، 345 و 543، 565.5) درجة مئوي . أظهرت متراكبات الايبوكسي/ الياف الكاربون موصلية حرارية أقل من متراكبات الايبوكسي/ الياف الكاربون المطلية بالنحاس. بعد المعالجة الحرارية، تم تحسين الاستقرار الحراري والتوصيل الحراري لمتراكبات الايبوكسي/ الياف الكاربون المعالجة. زاوية التماس لالياف الكاربون انخفضت قليلاً بعد المعالجة الكيميائية إلى 105 درجة. بعد الطلاء الكهربائي بالنحاس، أظهرت ألياف الكربون 82 درجة من زاوية التماس. أدت المعالجة الحرارية إلى تحسين قابلية البلل بشكل كبير لالياف الكاربون المطلية بالنحاس حيث تظهر 32 درجة من زاوية التماس عند 250 درجة مئوية.

Introduction

Carbon fiber/polymer composite (CFPC) materials are used in many technological industries because of their low density, high strength to weight ratio and high stiffness [1].

Many of the research conducted in the field of improving the properties of composites focused on improving the properties of the base material (matrix) [2]. However recently, research has been directed to improve the properties of the reinforcement phase in order to improve toughness, heat transport and interfacial adhesion between the matrix and fibers [3].

Metal-coated carbon fiber can be used for this purpose to improve wettability, to increase the lifetime of a part by increasing heat transport, to reduce hot spots and to reduce thermal stress by dispersing the heat [4].

Metal coating can be applied in different ways, such as electrode position, thermal evaporation, dipping and electroless plating [5, 6]. Electroless plating has been recognized as one of the most attractive and best ways to apply coatings on none or semiconducting surface. It is a process of deposing thin layer of metals, salts, oxides or any other compounds used in various industrial and technological applications. A redox reaction between a metal salt and a formaldehydes reduction in the same solution, a metal coating on surface can be formed in the absence of external current flow [5].

Over recent years, many researchers focused on using copper for coating fiber, glass and polymers due its excellent thermal conductivity and it's have the functional hydroxyl and carboxyl groups [7].

In many work, catalyst metal seeds such as platinum or palladium, which are relatively of high cost of production, are attached to the surface to cause the activation of the surface [8, 9]. However, the activation in this work was done using silver which is of low cost of production, and an attempt to make of pre-treatment (sensitization and activation) onto the surface of carbon fiber make the activation of the surface be successful.

Experimental work

Distilled water and acetone were used to clean the mat of carbon fiber, which was etched with sulfuric acid (3M H_2SO_4). The carbon fiber was sensitized using tin chloride solution containing (44 g $SnCl_2$+ 77 ml (3M HCl)), dried at 35 ^oC for 15 min and activated using silver nitrate solution containing (20gm $AgNO_3$ + 50ml (3M HCl)).

The copper plating solution was prepared by dissolving (1.3 gm copper sulfate as the main salt in 20 ml of distilled water) and stirred for 20 min to which a complex agent solution (2.6 g of Ethylenediaminetetraacetic acid disodium salt (Na_2EDTA) + 20 ml of distilled water) was then added. This was followed by adding sodium hydroxide solution (1.5gm NaOH+ 30 ml of distilled water) and 15 ml of Hydrazine monohydrate (N_2H_4-H_2O) was added drop wisely. 0.1 ml of Formaldehyde was then added as a reducing agent. Finally, the remaining 40 ml of distilled water was added.

The carbon fibers mat was kept in the prepared solution (of pH approximately equal 12). After chemical plating, the carbon fibers were placed in an oven for drying and heat treating at (150, 200, 250) ^oC for 1 hour. Table 1 shows the function of used material.

Table 1: List of materials used and its function.

Materials		**Function**
Composites	Carbon Fiber	Reinforced material
	Epoxy	matrix
Carbon fiber chemical treating solution	sulfuric acid (H_2SO_4)	etching
	tin chloride ($SnCl_2$) HCl	sensitization
	silver nitrate ($AgNO_3$)	activation
	HCl	
Copper chemical plating path	(copper sulfate $CuSO_4$)	Cu metal source for plating
	disodium ethylenediamine tetra acetate (EDTA-2Na)	complexing agent
	sodium hydroxide (NaOH)	PH adjuster
	Hydrazine monohydrate (N_2H_4-H_2O)	Stabilizer
	(Formaldehyde HCHO)	Reducing agent

The composite was prepared by Hand layup process. Initially, one layer of carbon fiber mat was placed in a mold where a thin layer of anti-adhesive coat is applied for easy extraction. The Epoxy resin material is applied using a brush on a reinforcement material. The composite's specimens were cured at room temperature for 24 hr to reach complete hardening. After that the composites were kept in the oven to complete curing at 70 $^{\circ}$C.

Thermal stability of the composites was evaluated with a thermogravimetric analyzer (TGA) (STA PT1000). The samples were heated from 0 $^{\circ}$C to 700 $^{\circ}$C with a heating rate of 10 $^{\circ}$C/min.

Thermal conductivity coefficient was measured using a Lee's disk. The samples used for thermal tests were cut to the same diameter as the copper plates of 40 mm and to a thickness of approximately 30mm.

Contact angle measurements were performed with a Rame-Hart instrument. An epoxy droplet is injected on the CF surface, and high resolution (60 fps) video camera captures the image of the epoxy droplet, and then analyzes the images.

Results and discussion

The elemental composition of the deposit was verified by energy dispersive X-ray (EDX) spectroscopy Fig.1. In addition to the carbon peak originating from the carbon substrate only copper features were noticed, presumably formed as an artifact due to manipulating with the copper-coated CF [10].

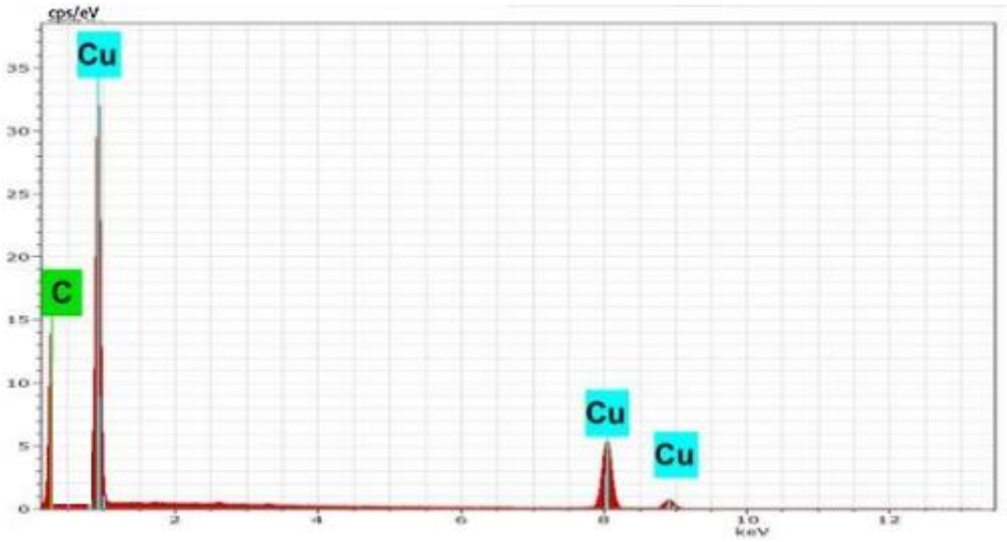

Fig.1: EDX spectroscopy of Cu coated CF.

Fig.2 and Table 2 shows the TGA results for Ep/CF, Ep/chemical treated CF and Ep/Cu coated CF composites at different treatment temperatures have a 5% weight loss (T_5) below 450 ˚C, due to the physically decomposition and adsorption of water [11, 12]. It can be noted that, for Ep/Cu coated CF, the temperature for the 5% weight loss increased to 322 ˚C when compare with that of the Ep/CF and Ep/chemical treated CF. This is due to the enhancement of interfacial adhesion after chemical treatment and Cu coating [12].

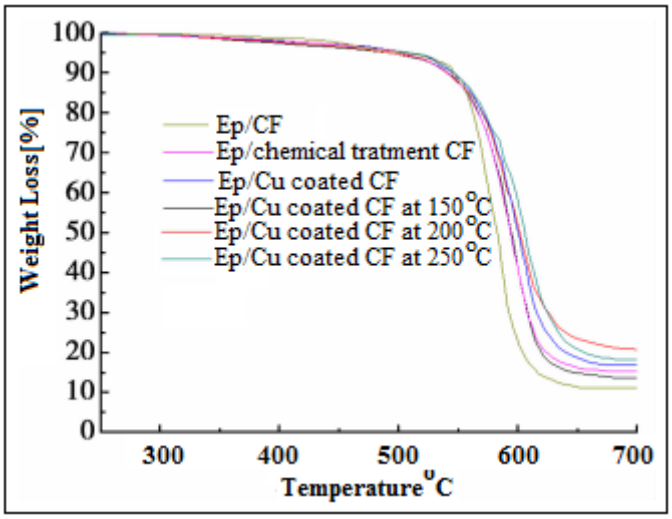

Fig.2: Thermal analysis data of Ep/CF, Ep/chemical treated CF and Ep/Cu coated CF composites at a different treatment temperatures (150, 200, 250) °C.

Table 2: Thermal analysis data of Ep/CF, Ep/chemical treated CF and Ep/Cu coated CF composites at different treatment temperatures (150, 200, 250) °C.

	T_g [°C]	T_5 [°C]	T_{10}[°C]	T_d[°C]
Ep/CF	76	246	266	522
Ep/chemical treated CF	83	298	310.3	543
Ep/Cu coated CF	88.5	322	345	565.5
Ep/Cu coated CF at 150°C	92	365.3	382	578
Ep/Cu coated CF at 200°C	96.2	392	409	591
Ep/Cu coated CF at 250°C	102.3	432	465	602

The temperature for 10 wt.% (T_{10}) and the degradation temperature (T_d) are obtained from Fig. 2 . As it can be seen, T_{10} and T_d of the Ep/CF are 266 and 522 $^{\circ}$C, respectively. With the chemical treatment and Cu coating, T_{10} and T_d of Ep/CF significantly increased to (310.3, 345 and 543, 565.5) $^{\circ}$C, respectively. Thermal stability of Cu coating CF was enhanced because of the thermal stability of Cu and the strong interaction bonds after chemical treatment. This agrees with the results of Bard et al. [11] and Mohd et al. [13].

TGA curves showed that the treatment temperature enhanced thermal stability of Ep/CF, this is due to the oxidation during heating [12].

Fig. 3 shows that Cu plating increased the composites conductivity which may be due to changes in contact resistance at the interface due to chemical modification and copper plating and tunneling resistance [12, 14]. The tunneling resistance between the Cu layer and CF is much smaller than that between copper and epoxy matrix. This effect, then leads to enhancing the thermal conductivities of the coated CF/Ep composites [14].

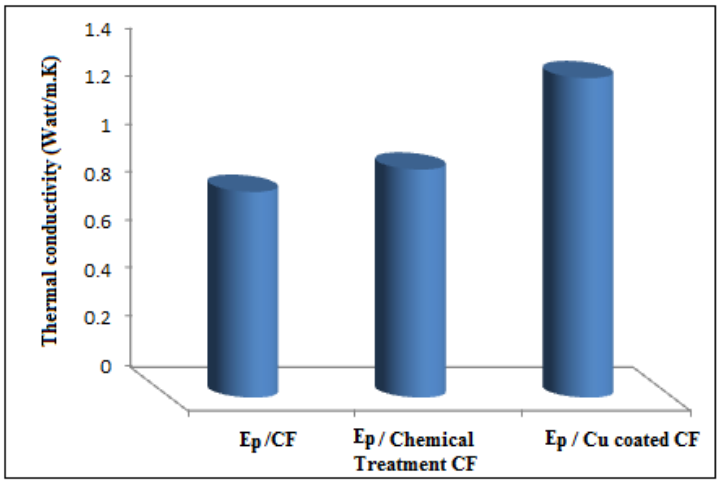

Fig. 3: Thermal conductivity of different type of Ep/CF composites.

Fig.4 shows that the thermal conductivity increases as the treatment temperature increase. This behavior is due to the increase of the deposition rate of copper as the temperature increases, which is due to a uniform and continuous dispersion with higher mechanical and chemical bonds [14, 11].

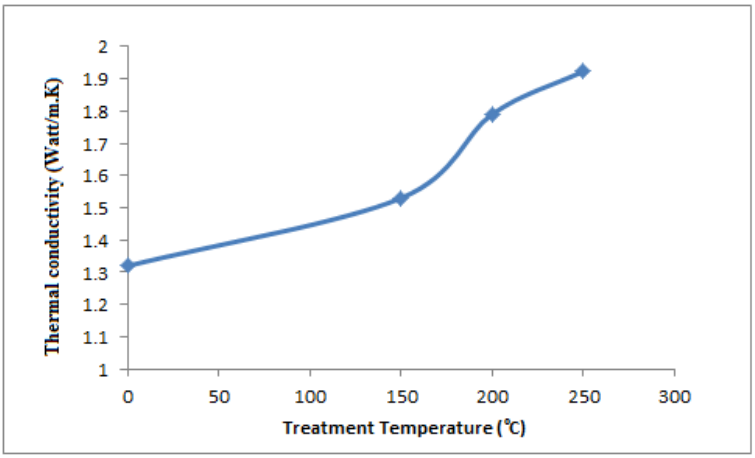

Fig.4: Thermal conductivity of Ep/Cu coated CF composites at different treatment temperatures (150, 200, 250) ^{o}C.

Fig.5 shows the contact angle of an epoxy drop on Ep/CF, Ep/chemical treated CF and Ep/Cu coated CF composites at different treatment temperatures.

The contact angle slightly decreased after chemical treatment to 105°. This is due to the change in the surface morphology and chemistry during the chemical etching which led to a more hydrophohilic behavior. It seems likely that the specific contact surface increased due to material removal which had a great effect on the contact angle [15].

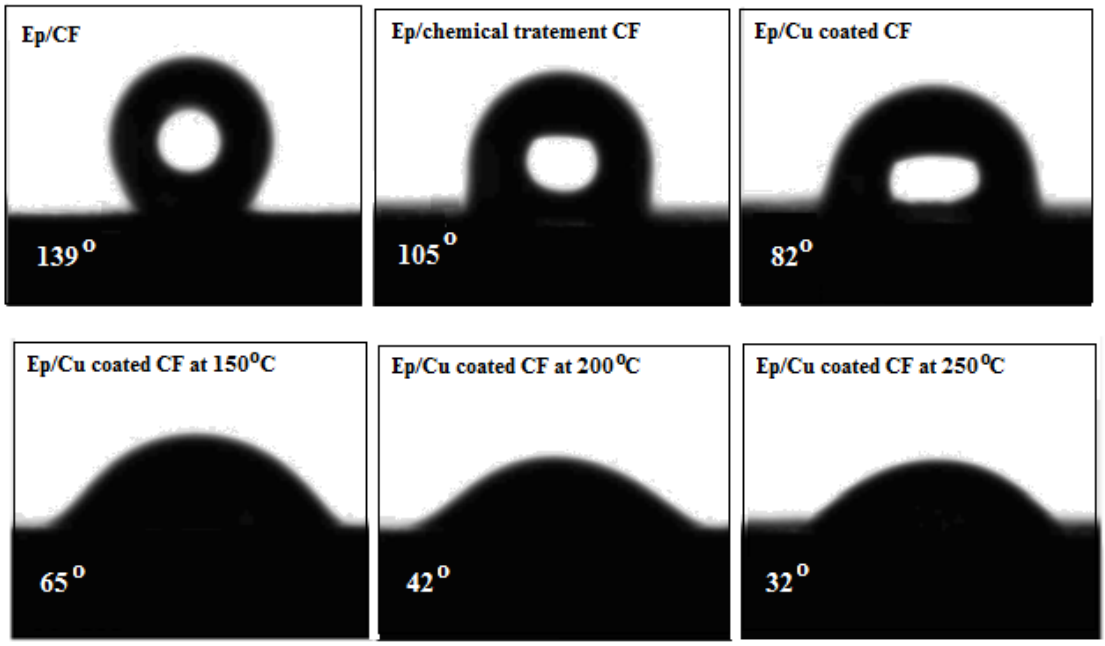

Fig. 5: Contact angle of Ep/CF, Ep/chemical treated CF and Ep/Cu coated CF composites at a different treatment temperatures (150, 200, 250) °C.

Coating the carbon fiber with Cu greatly improved the epoxy affinity and hence the surface energy. After electroless plating, the carbon fiber showed 82° contact angle, which indicates the increase of surface polarity caused the remaining wetting capability of the surface [16].

Heat treatment significantly improved wettability to a strong hydrophilic surface showing 32° of contact angle at 250 °C. Also it caused the increase of oxidation of a metal causing the rapid contact angle to decrease and this can strongly enhance wettability [16, 17].

Conclusions

The most critical problem of using fiber in the polymeric systems for electronic applications is compatibility, the interface control between them and the problems of thermal dissipation.

In this work, carbon fiber was modified with metal (Copper), since copper has high surface energy which allows extensive wetting with fiber and matrix. On the other hand, the high thermal conductivity of copper may increase the heat dissipation of composites.

The carbon fiber was etched by using H_2SO_4. Tin chloride and silver nitrite solution were used to pre-treat (sensitized and activated) the carbon fiber surface, respectively, then an electroless plating method was used to apply copper (Cu) on the surface of carbon fiber.

The thermal conductivity, thermal stability and wettability of prepared Cu modified CF/ epoxy composites were notice to improve at different treatment temperatures (150, 200 and 250) ^{o}C.

Acknowledgement

The auther's would like to express my special thanks to the material science lap staff in department of physics, college of science, university of Baghdad.

References

[1] F. Zhou, J. Zhang, S. Song, D. Yang, C. Wang, Materials, 12 (2019) 1-12.
[2] M. Roy, P. Tran, T. Dickens, A. Schrand, J. Compos. Sci., 4, 1 (2020) 1-25.
[3] M. Sharma, Sh. Gao, E. Mader, H. Sharma, L. Wei, J. Bijwe, Compos. Sci. Techno., 102 (2014) 35-50.
[4] J. Li, X. Li, C. Fan, H Yao, X. Chen, Y. Liu, Coatings, 7, 94 (2017) 1-11.
[5] J. Ni, M. Yu, K. Han, Materials Science Forum, 898 (2017) 2205-2213.
[6] Y. Li, H. Zhang, Y. Feng, G. Peng, Chinese Optics Letters, 7, 2 (2009) 115-117.
[7] W. Su, L. Yao, F. Yang, P. Li, J. Chen, L. Liang, Applied Surface Science, 257 (2011) 8067-8071.
[8] H. Jaafar, B. Aldabbagh, Baghdad Sci. J., 16 (2019) 632-638.
[9] S. Juan, E. Gordo, A. Morales, F. Sirois, Coatings, 10 (2020) 1-16.
[10] D. Riman, Z. Bartosova, V. Halouzka, J. Vacek, D. Jirovsky, J. Hrbac, RSC Adv., 5 (2015) 31245-31249.
[11] S. Bard, F. Schönl, M. Demleitner, V. Altstädt, Polymers, 11 (2019) 1-13.
[12] K. Kuniya, H. Arakawa, T. Kanai, A. Chiba, Trans. Japan Instit. Metals, 28 (1987) 819-826.
[13] N. H. Mohd, M. Abu Bakar, W. L. Tan, N. H. H. Abu Bakar, J. Ismail, C. H. See, J. Nanomater., 2012 (2012) 1-11.
[14] S. Yu, K. Park, J. Lee, S. Hong, C. Park, T. Han, C. Koo, Macromolecular Research, 25 (2017) 559-564.
[15] L. Xiong, F. Zhan, H. Liang, L. Chen, D. Lan, J. Mater Sci., 53 (2018) 2594-2603.
[16] W. Hao, X. Yao, Y. Ke, Y. Ma, F. Li, J. Adhesion Sci. Techno., 27 (2013) 1012-1022.
[17] Z. Dai, B. Zhang, F. Shi, M. Li, Z. Zhang, Y. Gu, Appl. Surf. Sci., 257 (2011) 8457- 8461.