Advanced Materials Research Vols. 602-604 (2013) pp 53-56
Online available since 2012/Dec/13 at www.scientific.net

doi:10.4028/www.scientific.net/AMR.602-604.53

Characterization of the Interphase in Carbon Fiber/Epoxy Composites Using Force Modulation Atomic Force Microscope

Hao Xue[1,a], Hongjun Fu[1,b]

[1]School of Textile, Tianjin Polytechnic University, Tianjin, 300387, China

[a]xuehao000@126.com, [b]fuhongjun@tjpu.edu.cn

Keywords: Atomic Force Microscope, Carbon Fiber, Epoxy, Interphase.

Abstract. In order to describe directly interphase properties of composite, force modulation of atomic force microscopy is adopted to study the cross-section of unidirectional carbon fiber/epoxy composites systematically. Research results indicate that in force modulation mode of AFM, relative stiffness of various phases distinct in distribution, which is described by probability histogram of relative stiffness. By comparison of probability histogram of relative stiffness nearby interphase of untreated and oxidation treated by ozone composites, the relative stiffness change oxidation treated one is more obviously to be found than the one untreated. Indirect show that obvious interphase formed by oxidation treatment. This method plays a valuable role in assessment of interphase strength of carbon fiber/epoxy, as well as in instruction composite production technology.

Introduction

The interphase exists from some area around the fiber surface where the chemical and mechanical properties begin to change from the matrix properties. An appropriately engineered interphase can significantly improve the strength of composites, which is a crucial constituent part of composites. Although the importance of the interphase has been widely accepted, up to now only few experimental means of establishing the interphase properties and it is an effective means of use atomic force microscope.

The atomic force microscope (AFM), invented by Binning etc, has developed into an excellent tool to obtain surface topography and interphase of many different types of material systems[1-3]. In contrast with STM, however, AFM with its resolution of atomic level is widely used in application of scientific research, not only because it is simple and eficient in sample preparation but also it does not need higher conductivity of material. To meet the needs of various physical proterties, different testing methods such as tapping mode, force modulation microscopy[4-5], atomic force acoustic microscopy or magnetic force modulation microscopy have been developed.

In this paper, the force modulation mode was adopted to study the interphase of unidirectional carbon fiber/epoxy composites. In combination with physical tests related to mechanical properties, interlaminar shear strength and stiffness of various phases are anylised and compared, targeting to composite untreated and treated with ozone oxidation.

Experimental

Materials and Equipments. The carbon fibers used in the experiments were 12K polyacrylonitrile based fibers manufactured by Toho Rayon Co., Ltd. The epoxy used was WSR6101 manufactured by Wuxi Resin Factory of Blue Star New Chemical Materials Co., Ltd. The ozone generator type was DHX-SS-1G manufactured by Harbin JiuJiu Electrochemical Engineering Technology Co., Ltd. The universal testing machine used was Instron 3369 manufactured by Instron corporation. An AFM(Benyuan cspm-5500) was used in the experiments.
Ozone oxidation treatment. Preparation ozone by ozone generator. Put extracted carbon fibers in glass capsule full of ozone and keep it in 80℃ water for 3 minutes.
Preparation of Samples. Unidirectional carbon fibers reinforced epoxy composites were prepared by molding method, controlling the mass fraction of fiber in 70%.

Force modulation microscopy. The force modulation microscopy, which can acquire morphology image and relative stiffness image, is extraordinary constituent part in AFM. Its principle of operation is that: Add extra electricity signal to the sample so that can vibrate vertically when using contact mode scan sample. Various stiffness of different area has different impact on deflection of cantilever (shown in Fig. 1). By way of detecting and recording the cantilever bending changes can reflect the relative stiffness distribution of the surface of the sample[6]. In view of them having distinct stiffness from different phases, this method can offer foundation of analysis mechanical properties of interphase modification.

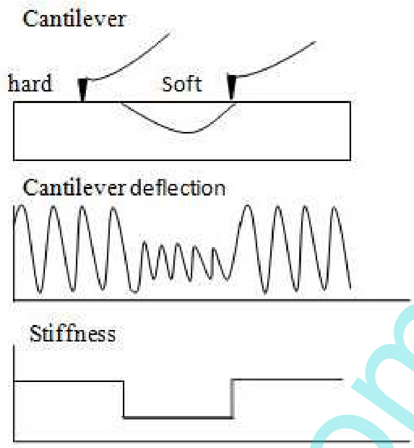


Fig. 1 Schematic diagram of atom force microscopy in force modulation mode

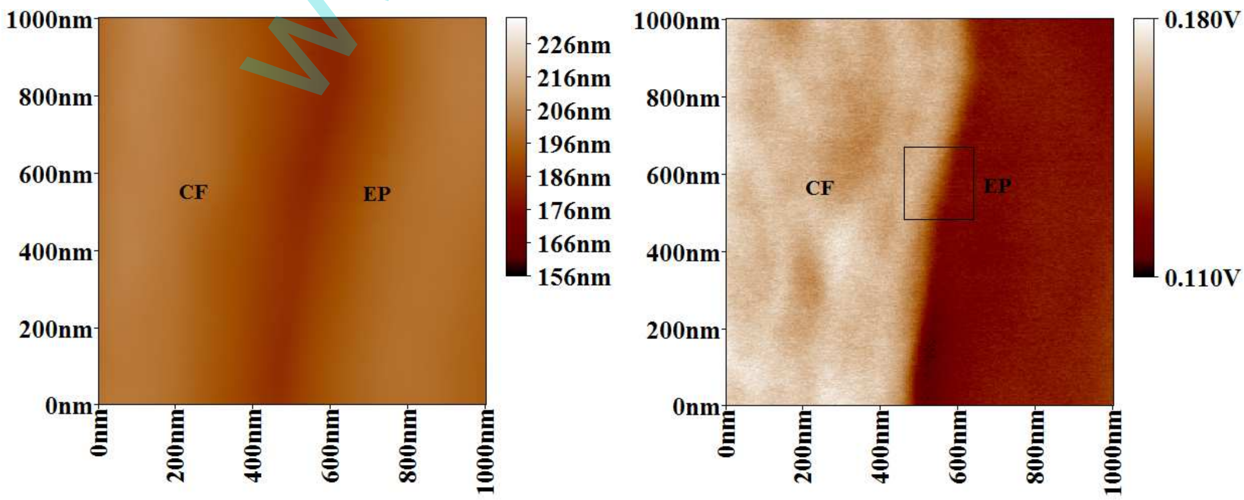
AFM test. AFM images on Benyuan CSPM-5500 operated in force modulation mode. The cantilever length 125um with spring constants 40N/m. Images consisted of 512×512 pixels, and the scanning frequency was 1 Hz. Image analysis was performed using the CSPM Imager software.

Results and discussion

Table 1. Results of oxidation treatment

Treatment Method	Untreated	Oxidation treated
ILSS/MPa	63.75	73

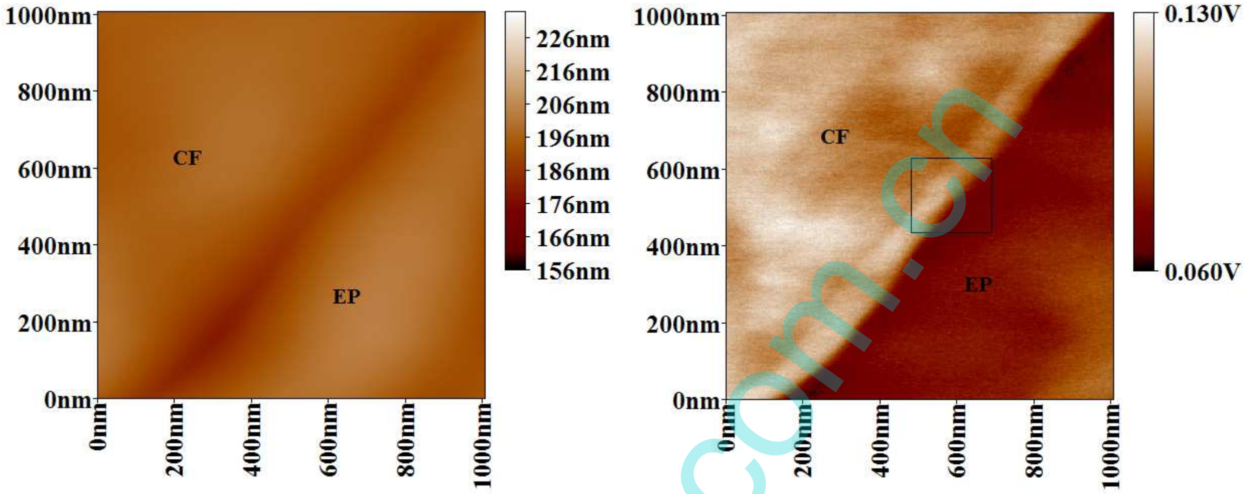
Measure the interlaminar shear strength of the composite samples on a universal testing machine using the three-point short beam bending method as specified in ASTM D 2344. It is found from Table 1 that ILSS of composite at 63.75MPa of untreated increase to 73MPa with oxidation. It shows that interphase strength can be improved dramatically by fiber surface ozone oxidation.



(a) Topography image (b) Relative stiffness image

Fig. 2 Topography and relative stiffness images of untreated CF/ EP composite surface

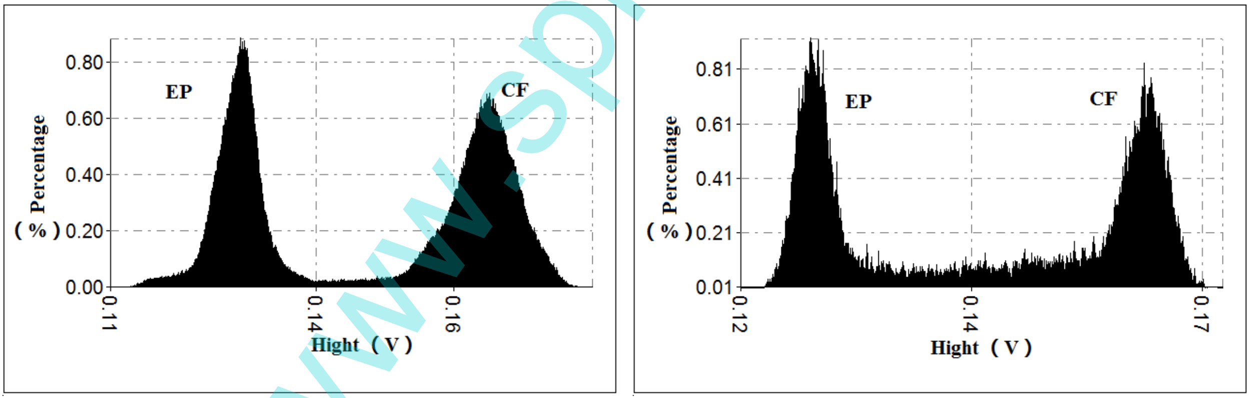
The Fig. 2 and Fig. 3 show the topography and relative stiffness images of cross-section areas of carbon fiber/exposy composite untreated and oxidation treated. It demonstrates in Fig. 2(a) and Fig. 3(a) that topography image of carbon fiber and epoxy composite shows little distinction, but the relative stiffness image displays a clear distinction between the carbon fiber and the surrounding epoxy in Fig. 2(b) and Fig. 3(b). A clear distinction can be observed between carbon fiber and epoxy in the relative stiffness images, the reason is that the relative stiffness image indicate surface stiffness distribution of material in the force modulation mode, and the stiffness difference between the carbon fiber and epoxy significantly more than the topography differences, utilizing stiffness diagram to observe the distribution of the two phases will be more clear.



(a) Topography image (b) Relative stiffness image

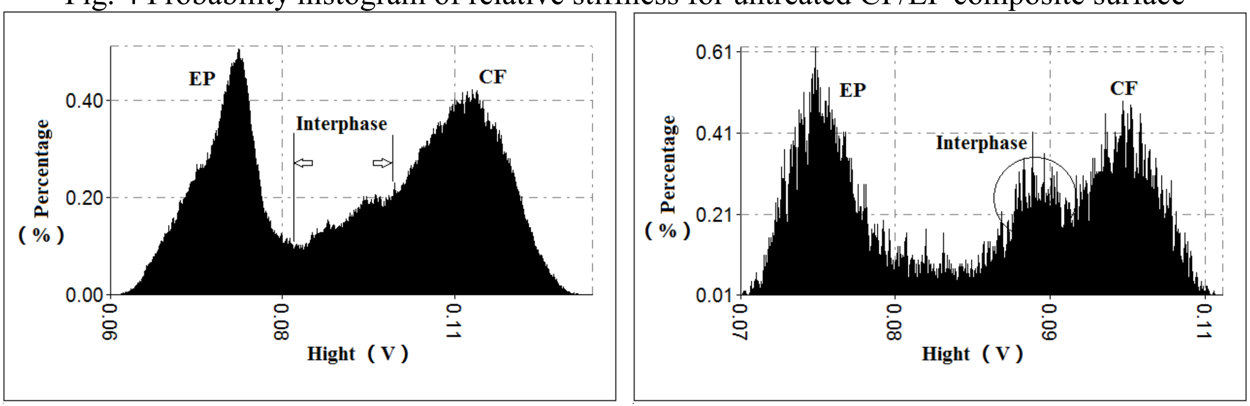
Fig. 3 Topography and relative stiffness images of oxidation treated CF/ EP composite surface

Probability histogram of relative stiffness as Fig.4 and Fig.5 as follows can be acquired by CSPM Imager software.



(a) (b)

Fig. 4 Probability histogram of relative stiffness for untreated CF/EP composite surface



(a) (b)

Fig. 5 Probability histogram of relative stiffness for oxidation treated CF/EP composite surface

The abscissa denotes the relative stiffness values(indirectly indicated by voltage generate from the cantilever deflection), and the vertical axis denotes the percentage of the relative stiffness values. It can be seen that the stiffness distribution curve clearly into two peaks in Fig. 4(a), obviously indicating that there are two stiffness phases on the surface, the stiffness of carbon fibers is higher than epoxy. Regarding nanometer leveled thickness of composite interphase, which might possibly be neglected in broad relative stiffness image, so we select smaller locations near the interphase of the material and make several statistical analyzes. The probability histograms of relative stiffness is shown in Fig. 4(b), there are only two distinct stiffness peaks, and no obvious transition between the two peaks. It follows that no obvious interphase in composite materials without fiber surface modification.

According to the probability histograms of relative stiffness as shown Fig. 5(a), it seems to have a new stiffness phase between carbon fibe and epoxy appeared. Selected the appropriate locations near the interphase and made statistical analysis, as shown the stiffness distribution histogram in Fig. 5(b). It can be seen that the transition is no longer clear and intense between carbon fibe and epoxy. It manifests that there are some changes on the interphase of composite after fiber surface modification. This evidences that interphase formation signally after fiber treated with oxidation. The reasons for this condition is that: weak surface layer was removed and covered by more oxygen-containing groups generated on carbon fibers surface after oxidation process, which results in improvement of infiltration of fiber and resin, so as to strengthen the composite interphase.

Conclusions

1) Images of topography and relative stiffness of composites can be obtained simultaneously by adopting force modulation mode of AFM. The relative stiffness images reflect more obviously the material distribution, and stiffness changes.
2) Relative stiffness of fiber, matrix and interphase can be quantitively characterized by probability histogram of relative stiffnes. Small area close to interphase should be selected in order to observe clearly interphase phases in the histogram.
3) Composites have no distinct interphase if fiber surface is not treated. Interphase structure becomes complicated after oxidation treatment, as moderate modulus transition is formed between fiber and resin, which significantly improves the mechanicial properties of material interphase.

Acknowledgments

The authors would like to thank the National Natural Science Foundation of China for financial support (No. 51003075).

References

[1] Chunli Bai, Fang Tian, Ke Luo. Scanning Force Microscopy. Science Press, 2000, in Chinese.

[2] Yingge Yang, Hai Zhou, Yimin Lu. Microtechnique & Measurement, Vol. 5 (2008), p. 68-71

[3] Xugang Yang, Xiao Yang. Atomic Force Microscopy and Its Application. Chemical Industry Press, 2012, in Chinese.

[4] P Maivald, H J Butt, S A C Gould, C B Prater, B Drake, J A Guriey, V B Elings, and P K Hansma. Nanotechnology, Vol. 2 (1991), p. 103-106.

[5] Hongjun Fu, Yudong Huang, Li Liu. Acta Materiae Compositae Sinica. Vol. 22(2005), p. 85.

[6] M. Radmacher. R. W. Tillmann, and H. E. Gaub. Biophysical Journal, Vol. 64(1993), p. 735-736.