Influence of rare earth surface treatment on tensile properties of aramid fiber reinforced epoxy composites

JU WU, XIANHUA CHENG*, CHAOYING XIE
School of Mechanical Engineering, Shanghai Jiao Tong University, Shanghai 200030, People's Republic of China
E-mail: Xhcheng@mail.sjtu.edu.cn

Structures made of aramid-fiber-reinforced (ARF) plastics are widely used in aviation and space engineering due to the low density and high specific strength of aramid fibers and composites based on them, which is important for the aircraft industry [1]. The interfacial adhesion of fiber reinforced composites plays a very important role in determining the composite mechanical properties. A better fiber/matrix interfacial adhesion/bond will enhance properties such as tensile strength, interlaminar shear strength, delamination resistance, fatigue and corrosion resistance [2]. However the surface of aramid fiber is chemically inert and smooth, and its compatibility with matrix resin is bad, thus its adhesion with the resin matrix is poor. Therefore, to use aramid fiber as reinforcement, surface-modification is essential to enhance its reinforcing effect [3].

There are several fiber surface modification methods, such as chemical treatment (including coupling agent and chemically grafting methods) and plasma treatment used to improve the adhesion with the resin matrix [4].

Rare earths (RES) surface treatment had been applied to modify the surface of inorganic fiber (such as glass fiber) [5, 6], and an improvement of interfacial adhesion between glass fiber and resin was achieved, but this treatment has never been tried for organic fiber surface treatment. RES surface modification method has some virtues, such as no pollution to environment, low cost, high efficiency, simple process and no damage to fiber. In the present research, a solution with rare earths was used to modify the surface of aramid fibers and compared with the epoxy chloropropane (ECP) grafting modification method [7]. Tensile properties of both the aramid/epoxy composites and single fiber were determined. The effects of RES concentration on tensile properties of composites were investigated in detail in order to explore an optimum amount of rare earth in solution for modifying aramid fiber.

Aramid fibers used in this study were F-12 aramid fibers. Rare earth compound $LaCl_3$ purchased from Shanghai Yuelong New Materials Co., Ltd. was used as main component of rare earths applied in surface modification solution. E-51 epoxy resin and 593 curing agent were manufactured by Shanghai Resin Factory Co., Ltd. and epoxy chloropropane (ECP) was produced by Shanghai Yonghua reagent factory.

Before surface treatment, F-12 aramid fibers were circumfluent extracted by toluol, acetone and deionized water for 3 h in sequence, then dried in a vacuum oven at 110 °C for 3 h.

Two types of fiber surface treatment have been applied in this research: RES treatment and ECP grafting modification treatment.

For ECP grafting modification treatment, F-12 aramid fibers were immersed in the solution of KOH (0.7%)/alcohol for 2 h, then washed and dried. After that, these fibers were grafted in ECP at 90 °C for 6 h, then washed with distilled water and dried.

For RES surface treatment, F-12 aramid fibers were immersed in the RES/alcoholic solution at room temperature for one hour, and dried in a vacuum oven at 110 °C for 4 h. The $LaCl_3$ content in alcoholic solution was from 0.1 to 0.9 wt%.

F-12 aramid/epoxy unidirectional laminated composites were manufactured. The content of F-12 aramid fibers was fixed at 60% by volume for all composite specimens. The ratio of E-51 epoxy resin to 593 curing agent was 100:25 in weight. This mixture can be cured at room temperature. The tensile specimens were prepared according to Chinese national standards (GB3354-82), with a size of 230 mm $\times$ (12.5 $\pm$ 0.5 mm) $\times$ (2 $\pm$ 0.3 mm). The tensile properties of composites were tested by AG-100kNA material tensile machine, at a cross-head speed of 5 mm/min. An average value was obtained from lists on 5 specimens for each experimental data.

Single aramid fiber tensile specimens were prepared by attaching a single fiber to a paper frame using epoxy resin. It was tested using an Instron Tensile Testing Machine (Model 2211), at a cross-head speed of 2 mm/min.

Fig. 1 gives the tensile strength and tensile modulus of RES treated aramid/epoxy composite as a function of RES concentration. It is seen that the tensile strength and tensile modulus increases with RES concentration when RES concentration is less than 0.5 wt%. The maximum values of tensile strength and modulus were obtained at 0.5 wt% RES concentration. Above the maximum value, the tensile properties decreased gradually. According to the chemical bonding theory, it is suggested that rare earth is absorbed onto the aramid fiber surface through chemical bonding, which increases the concentration of reactive functional groups due

*Author to whom all correspondence should be addressed.

0022–2461

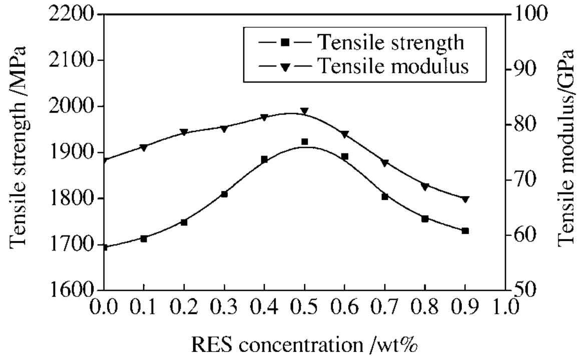


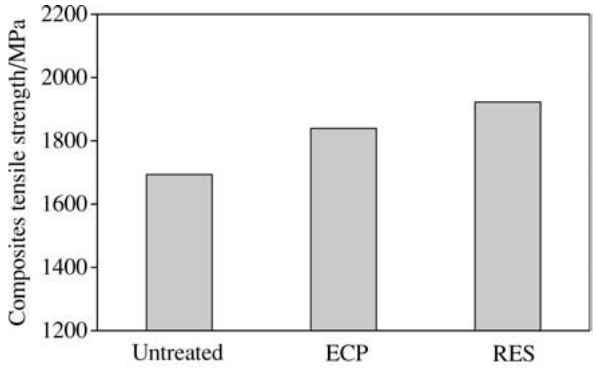
Figure 1 Relationship between tensile properties of RES treated aramid/epoxy composites and RES concentration.

to the chemical activity of rare earth element. These reactive functional groups can improve the compatibility between aramid fibers and epoxy matrix and form a chemical combination between aramid fiber and epoxy matrix. As a result, the interfacial adhesion of aramid/epoxy composite is improved through RES surface treatment. However, excess rare earths may result in the formation of rare earth salt crystals on aramid fiber surface, consequently, a decrease of the tensile properties of aramid/epoxy composite occurred. The interfacial changes are still under investigation.

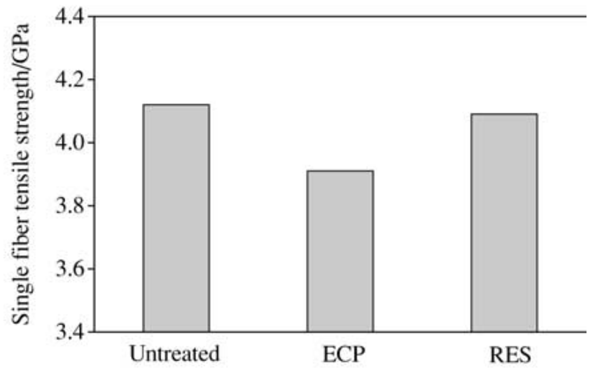
According to above tensile experimental results, the concentration of RES was fixed at 0.5 wt%, while a comparison between RES treatment and ECP grafting treatment was made as shown in Fig. 2. It is seen in Fig. 2a that both surface treatments can improve the tensile properties of aramid/epoxy composite, and RES treated aramid fibers yielded better results. The tensile strength of RES treated aramid/epoxy composite has been improved about 13.5 percent compared with that of untreated composite. And 8.6 percent improvement was achieved by ECP grafting treatment.

From single fiber tensile test results shown in Fig. 2b, it is clear that ECP grafting treatment does more damages to aramid fibers. The tensile strength of F-12 aramid single fibers treated with ECP grafting method decreases about 5.1 percent compared with that of untreated aramid fibers, which is in accordance with the results in [7]. It is noticed that during ECP treatment aramid fibers were treated with KOH/alcohol solutions to introduce —COOK groups to the surface of aramid fibers as grafting initiators, which may lead to hydrolyzation of aramid fiber surface molecules. Excess hydrolyzation may result in damage to fibers and, then, affect the tensile properties of composites, as shown in Fig. 2b. Whereas the tensile properties of RES treated aramid fibers are not changed obviously, which reveals little damage done by RES treatment to F-12 aramid fibers.

In conclusion, RES treatment is superior to ECP grafting treatment in promoting interfacial adhesion between F-12 aramid fiber and epoxy matrix. The tensile properties of RES treated F-12 aramid/epoxy composites are affected by the overall rare earths concentration in alcoholic solution. The tensile properties of the F-12 aramid/epoxy composite can be improved considerably when the rare earths concentration is 0.4–0.6 wt%. The optimum amount of RES is 0.5 wt%.



(a) Effect of surface treatments on tensile properties of F-12 / epoxy composite



(b) Effect of surface treatments on tensile properties of F-12 fiber

Figure 2 Effect of surface treatments on tensile properties of F-12 fiber and its epoxy composite.

Acknowledgments
Project supported by National Natural Science Foundation of China (No. 50275093) and Astronautics Supporting Technology Foundation (2001-HT-SHJD).

References
1. T. A. MAN'KO, E. A. DZHUR, F. P. SANIN and I. M. ERMOLAEV, *Mech. Compos. Mater.* **37** (2001) 171.
2. B. C. RAY, *J. Mater. Sci. Lett.* **22** (2003) 201.
3. JENG-SHYONG LIN, *Europ. Polym. J.* **38** (2002) 79.
4. R. J. YOUNG, D. J. BANNISTER, *et al.*, *J. Mater. Sci.* **35** (2000) 1939.
5. YUJUN XUE and XIANHUA CHENG, *J. Mater. Sci. Lett.* **20** (2001) 1729.
6. X. H. CHENG and Y. J. XUE, *J. Rare Earths.* **19** (2001) 238.
7. LONG JUN, ZHANG ZHIQIAN, *et al.*, *Mater. Sci. Techn.* **7** (1999) 46.

Received 29 May
and accepted 7 August 2003