Effects of sheet electron beam irradiation on aircraft design stress of carbon fiber

YOSHITAKE NISHI, AKIHIRO MIZUTANI, ATSUSHI KIMURA, TAKASHI TORIYAMA, KAZUYA OGURI, AKIRA TONEGAWA *Department of Materials Science, Tokai University, 1117, Kitakaname, Hiratsuka, Kanagawa, 259-1292 Japan E-mail: am026429@keyaki.cc.u-tokai.ac.jp*

Using sheet electron beam (EB) irradiation, reinforcement for carbon fiber was achieved. In order to annihilate twisting strain in carbon fiber, the fracture stress was precisely obtained by means of a twisting free tensile test developed. The EB treatment enhanced the fracture stress at different integrated fracture rates (*R*f) and increased Weibull modulus. It also enhanced design stress, when the integrated fracture probability (P_f) is 10⁻⁵ for aircraft materials. The aircraft design stress (5 GPa) was approximately 4 GPa larger than that (1 GPa) before EB treatment. ^C *2003 Kluwer Academic Publishers*

1. Introduction

Carbon fiber composites have been applied as highstrength light structural materials in the fields of aerospace and rapid transit engineering [1, 2]. Previous studies have established the fracture stress of carbon fiber to be approximately 6 GPa up to 20th century. Heat treatment has been used to enhance the fracture stress of carbon fibers [3], because carbon atoms diffuse and rearrange near cracks at high temperatures. If a crack reduction treatment can be performed without heat, the strength of carbon fiber composite materials will increase while production costs are lowered, allowing the use of such strengthened structural materials for aerospace and rapid transit applications. In order to improve material fracture strain [4] and fracture stress [5] at lower temperatures, treatment by electron beam irradiation has been explored. The EB treatment has succeeded in obtaining high fracture strain for slight EB irradiation below 5 Mrad, using an integrated treatment term of 0.26 sec [4], and has also achieved high fracture stress for short term EB irradiation below 50 Mrad [5]. Furthermore, 10 GPa class carbon fibers have been recently obtained by longer-term EB-irradiation from 6.5–9.6 sec of integrated treatment. Here we report the reinforcement method of carbon fibers by using sheet electron beam treatment and its design stress, when the integrated fracture probability (P_f) is 10⁻⁵ for aircraft materials.

2. Experimental

The carbon fiber $(6 \times 10^{-6} \text{ m}$ in diameter, Filament 12000f ASAHI-NIPPON CARBON FIBER Co. Ltd. TOKYO JAPAN) was cut into pieces 50 mm in length. The sheet electron beam irradiation was homogeneously performed using an electron-curtain processor (Type CB175/15/180L, Energy Science Inc., woburn, MA) [4–8]. The acceleration potential and the irradiating current densities were 170 kV and 0.089 A/m^2 , respectively. The sheet electron beam irradiation treatment was applied intermittently (i.e., not continuously). The conveyer speed was 10 m/min. Irradiation time was kept constant at 0.23 s in order to control the temperature in each of the samples. The temperature of the sample was below 323 K just after the irradiation. The irradiation dose was controlled by the integrated irradiation time in each of the samples. Here, the total amount of absorbed dose value was converted by the absorbed dose of the distillation water. Although the EB generation was in vacuum, the irradiated specimen was kept under protective nitrogen at atmospheric pressure in the apparatus. Namely, the specimen was irradiated by electron beam through the titanium thin film window attached to the vacuum chamber (240 mm in diameter). The distance between sample and window is 35 mm. The oxygen concentration was less than 400 ppm in this atmosphere.

In order to evaluate fracture stress, a twisting free tensile test was developed, as shown in Fig. 2. The water dropping method was used to apply the load, providing an untwisting stress to the carbon fiber loaded samples. The water was dropped into micro-cell from a capillary of cotton fiber flux (see Fig. 2). The stress rate was 0.3 N/s. To confirm the test system, the fracture stress of the commercially used carbon fibers 'TORAYCA-T800H' (Toray Ltd. Tokyo) was obtained at different fracture probabilities. The fracture stress ranged from 3.7–5.3 GPa, while the Weibull coefficient (m) was found to be 9.2.

3. Results

To evaluate fracture resistance, an integrated fracture rate (R_f) is expressed by the following equation.

$$
R_{\rm f} = I/n \tag{1}
$$

Figure 1 Schematic drawing of sheet electron beam irradiation performed using an electron-curtain processor (Type CB175/15/180L, Energy Science Inc., Woburn, MA).

Figure 2 Schematic drawing of a twisting free tensils test to evaluate tensile strength on a fracture. Samples were loaded by water dropping method to annihilate twisting stress of carbon fiber. The water was dropped into micro-cell from capillary of cotton fiber flux. The stress rate was 0.3 N/s.

Here, *n* and *I* are total sample number and fractured order of each sample, respectively. Changes in the fracture stresses of the carbon fiber samples in the tensile test are plotted against electron beam irradiation dose at different integrated fracture rates (R_f) in Fig. 3, indicating that a 10 GPa class carbon fiber can be obtained with 112 and 151 Mrad-irradiations. The integrated fracture rates (R_f) increased with increasing fracture stress (σ_f) . In order to enhance the fracture stress, the EB treatment has been developed for carbon fiber by using sheet electron beam irradiation. Namely, it enlarged the fracture toughness for carbon fiber. The EB treatment below 112 Mrad-irradiation enhances fracture stress (σ_f) of carbon fiber at different integrated fracture rates (R_f) , whereas the excess EB treatment above 200 Mrad irradiation cannot obtain 10 GPa class carbon fibers.

When the EB treatment below 112 Mrad was performed, the carbon fiber fracture stress was improved. The maximum value of fracture stress was found in the carbon fiber samples irradiated at 112 Mrad. Namely, the EB treatment increased the tensile strength of fracture. In order to get 10 GPa class carbon fibers, we

Figure 3 Changes in the fracture stresses of the carbon fiber samples in the tensile test against electron beam irradiation dose at different integrated fracture rates (R_f) , indicating that a 10 GPa class carbon fiber can be obtained with 112 and 151 Mrad-irradiations.

concluded that 6.5–9.6 sec of integrated EB-irradiation treatment should be applied near room temperature.

4. Discussion

4.1. Weibull coefficient and design stress for airplane

To evaluate fracture resistance, integrated fracture probability (P_f) was expressed by the following equation by using Median Rank method broadly applied [9].

$$
P_{\rm f} = (I - 0.3)/(n + 0.4) \tag{2}
$$

Here, *n* and *I* are total sample number and fractured order of each sample, respectively. The fracture probability (P_f) depended on risk of rupture (σ_f/σ_0) and was expressed by the following equation [10].

$$
P_{\rm f} = 1 - \exp[-(\sigma_{\rm f}/\sigma_0)^m]
$$
 (3)

 σ_f was the experimentally obtained fracture stress. When P_f was equal to 0.967, an ideal fracture stress (σ_0) can be obtained. A Weibull coefficient (m) indicated the statistical distribution of fracture stress. A linear relationship as shown in Fig. 4 was obtained for

Figure 4 Change in linear relationship of Weibull plots against fracture stress on tensile test for carbon fiber samples at different irradiation doses of sheet electron beam irradiation.

Figure 5 Change in Weibull modulus against irradiation doses of sheet electron beam irradiation for carbon fiber samples.

carbon fiber samples before and after EB treatment. Fracture stress values of the carbon fiber samples were distributed from 2–6 GPa and the Weibull modulus is about 3.2 before EB treatment. The treatment enhanced the Weibull modulus, as shown in Fig. 5. The EB treatment below 112 Mrad enhanced Weibull modulus from 3.2 to 8.0. Since the treatment using high electrical potential (170 kV) probably migrates terminated unstable carbon atoms in a carbon fiber, such migration probably served to annihilate vacant sites which might act as crack origins, to dull the edge of sharp crack tips and to relax the stress concentration. Therefore, the treatment of sheet electron beam irradiation decreased the statistical distribution of fracture toughness.

In order to design the aircraft, an aircraft design stress at 10−⁵ of integrated fracture probability (*P*f) is an important factor. Fig. 6 shows the change in designed fracture stress against irradiation dose, when integrated fracture probability (P_f) based on aircraft materials standard was 10^{-5} . The EB treatment enhanced the aircraft design stress. Although the design stresses were 1 GPa for carbon fiber before EB treatment, the design stress was 5 GPa at 10⁻⁵ of P_f for samples treated by EB treatment of 112 Mrad. The EB treatment enhanced design stress, for which the maximum value was approximately 4 GPa larger than that before EB treatment.

Figure 6 Change in aircraft design stress against irradiation doses of sheet electron beam irradiation for carbon fiber samples at 10−⁵ of integrated fracture probability (P_f) .

4.2. Discussion for fracture stress enhancement by SEBI treatment of carbon fiber

Based on the X-ray diffraction patterns before and after SEBI treatment [5], remarkable structure change cannot be found, as shown in Fig. 7. On the other hand, large shape change of stress–strain (σ - ε) curves for carbon fiber samples before and after the irradiation had been observed, as shown in Fig. 8. The initial $d\sigma/d\varepsilon$ value of the carbon fiber sample after the treatment was larger than that of the carbon fiber sample before the treatment. Thus, the existence of the structure change could be deduced. Carbon fiber used to involve vacant site. If the vacant site density decreased in carbon fiber, the density of dangling bonds should decrease. When the EB treatment forcibly diffused terminated unstable carbon atoms into vacant sites and incoherent interfaces because of high electrical potential of 170 kV, such carbon atom migration should decrease the dangling bonds. In order to evaluate the inter-atomic bonding density, the density of dangling bonds was obtained by dangling bond signals of graphite, which could be detected by means of electron spin resonance (ESR) spectroscopy [5]. The EB treatment decreased the density of dangling bonds [5]. Namely, since the EB treatment

Figure 7 X-ray diffraction patterns of carbon fiber samples before and after the 112 Mrad EB irradiation.

Figure 8 Stress–strain (σ - ε) curves for carbon fiber samples before and after the 112 Mrad EB irradiation.

forcibly diffused carbon atoms to vacant sites, incoherent interfaces and edges of sharp crack tips, it enhanced the aircraft design stress.

On the other hand, it can be explained that the excess EB treatment decreases the aircraft design stress (see Fig. 6), if the excess EB treatment forms interstitial atoms with dangling bonds between the hexagonal graphite planes.

5. Conclusion

Reinforcement has been developed for carbon fiber by using sheet electron beam irradiation. In order to evaluate fracture stress, precisely, a twisting free tensile test was also developed and performed to annihilate twisting stress in carbon fiber. The EB treatment up to 112 Mrad largely enhanced the Weibull modulus and the aircraft design tensile strength of the EB treated sample, which was approximately 4 GPa larger than that before EB treatment.

References

1. J. D. BROOKS and G. H. TAYLOR, *Carbon* **3** (1965) 185. 2. T. CHANG and A. OKURA, *Transactions ISIJ* **27** (1987) 229.

- 3. C. R. ROWE and D. L. LOWE, *Bienn. Conf. Carbon* **13** (1977) 170.
- 4. Y. NISHI, T. TORIYAMA, K. OGURI, A. TONEGAWA and K. TAKAYAMA, *J. Materials Research* **16** (2001) 1632.
- 5. A. KIMURA, A. MIZUTANI, TORIYAMA, K. OGURI, A. TONEGAWA and Y. NISHI, in Proceedings of the Sixth Applied Diamond Conference/Second Frontier Carbon Technology Joint Conference [ADC/FCT 2001], Auburn, AL, USA, August 4–10, 2001, p. 779.
- 6. Y. NISHI, S . TAKAGI, K. YASUDA and K. ITOH, *J. Appl. Phys.* **70** (1991) 367.
- 7. Y. NISHI, H. IZUMI, J. KAWANO, K. OGURI, Y. KAWAGUCHI, M. OGATA, A. TONEGAWA, K. TAKAYAMA, T. KAWAI and M. OCHI, *J. Mater. Sci.* **32** (1997) 3637.
- 8. K. OGURI, K. FUJITA, M. TAKAHASHI, Y. OMORI, A. TONEGAWA, N. HONDA, M. OCHI, K. TAKAYAMA and Y. NISHI, *J. Materials Research* **13** (1998) 3368.
- 9. T. NISHIDA and E. YASUDA, "Evaluation of Dynamic Properties of Ceramics" (in Japanese: Ceramics no Tokusei Hyouka) (Nikann Kougyou Shinnbunn, Tokyo, 1986) 50.
- 10. S. W. FREIMAN, "Glass: Science and Technology, Vol. 5, in Elasticity and Strength in Glasses," edited by D. R. Uhlmann and N. J. Kreidl (Academic Press, 1980) p. 63.

Received 13 November 2001 and accepted 8 July 2002