Effects of EB irradiation on spring constant of C/C composite coil

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In order to enhance a spring constant of C/C composite coil spring, the electron beam irradiation was homogeneously performed. The EB irradiation largely enhanced initial spring constants, which were higher than that before EB irradiation. The constant spring constant was also obtained by 150 Mrad-irradiation for the C/C composite coil spring. © 2003 Kluwer Academic Publishers

1. Introduction

C/C composites have been applied as high-strengthened light structural materials in the fields of aerospace and rapid transit engineering [1,2]. Furthermore, C/C composite coil spring shows high stress resistance to heat near 2000 K. However the C/C composite coil spring had two serious problems to apply precise engineering for initial strain range. One is its low spring constant. Another is a non-linear stress-strain curve of the spring. In order to enhance a spring constant and elastic linearity of C/C composite coil spring, the electron beam (EB) irradiation was performed. The EB irradiation of high electrical potential (170 kV) is used to migrate carbon atoms in a carbon fiber. Such carbon atom migration probably relaxed the residual stress [3–5]. Furthermore, the impurity atoms might also occupy the sites, simultaneously, because the EB-irradiation usually enhanced the surface activation of materials [5-8]. Namely, the EB-reinforcement may be caused by annihilations of stress relaxation. If the EB treatment can be also effective for C/C composite coil spring, high spring constant and linear elasticity relationship between stress and strain can be expected. Therefore, the purpose of the present work is to study effects of EB treatment on spring constant of C/C composite coil spring.

2. Experimental procedure

The shape of C/C composite spring (Dr. Takao Nakagawa, President, Across Ltd., 16–27, Nishiki-cho 2, Warabi, Saitama, JAPAN, E-mail:across@sannet. ne.jp) is shown in Fig. 1. The sizes were 50 mm in diameter and 25 mm in length. HIP treatment was

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performed for 7 h from 673 to 873 K under 11.8 MPa. After that, heat treatment was performed at elevated temperatures up to 2273 K under argon atmosphere. The heating rate was 20 deg/h.

The sheet electron beam irradiation was homogeneously performed using an electron-curtain processor (Type CB175/15/180L, Energy Science Inc., Woburn, MA, Iwasaki Electric Group Co. Ltd. Tokyo) [5-8], as shown in Fig. 2. The acceleration potential and the irradiating current density were 170 kV and 0.89×10^{-2} mA/cm², respectively. The EB 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 four samples. The temperature of the sample was below 323 K just after the EB 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 atmospheric pressure in the apparatus. Namely, specimen was irradiated by electron beam through the titanium thin film window attached to 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.

To evaluate compressive strength on elasticity, a compressive test was performed. The C/C composite spring on fixed stand was homogeneously compressed. Fig. 3 shows schematic drawing of compression test of C/C composite spring. The compressive stress rate was 3.3×10^{-2} N/s.



Figure 1 The shape of C/C composite spring (Dr. Takao Nakagawa, President, Across Ltd., 16-27, Nishiki-cho 2, Warabi, Saitama, Japan).



Figure 2 Schematic diagram of electron-curtain processor (Type CB175/15/180L, Energy Science Inc., Woburn, MA, Iwasaki Electric Group Co. Ltd. Tokyo).



Figure 3 Schematic drawing of compression test of C/C composite spring.

3. Results

3.1. Low initial spring constant of C/C composite coil

To evaluate the initial spring constant of C/C composite coil spring, the compressive test was performed below 15 kPa. Open circles in Fig. 4 show change in compressive stress against strain of C/C composite coil springs before EB treatment. Increasing the compressive strain enhanced the resistance to compressive stress. The initial compressive stress doesn't largely increase the



Figure 4 Change in compressive stress against strain of C/C composite coil springs before and after 150 Mrad-EB treatment.



Figure 5 Change in spring constant against compressive strain.

compressive stress below 0.12 of initial compressive strain for C/C composite spring before EB treatment. Namely, the dull slope above 0.12 of compressive strain was observed in the untreated C/C spring. Open squares in Fig. 5 show a change in spring constant against compressive strain. The spring constant is not constant below 0.20 of compressive strain. It shows that the C/C spring coil before EB treatment cannot be applied for precise mass balance.

3.2. EB-strengthening of C/C composite coil Open triangles in Fig. 4 show change in compressive stress against strain of C/C composite coil springs after 150 Mrad-EB irradiation. Increasing the compressive strain largely enhanced the resistance to compressive stress, as shown in solid line. The steep slope of curve was observed in all C/C composite coil springs treated by 150 Mrad-EB irradiation.

The compressive strain straightly increases the compressive stress for C/C composite spring after 150 Mrad-EB irradiation. Namely, the sharp slope was observed in the treated C/C spring. Open circles in Fig. 5 show a change in spring constant against compressive strain. The initial spring constant for the treated sample (150 Mrad) is about three times larger than that



Figure 6 Change in spring constant at 0.15 of strain against irradiation dose.

for untreated sample. Since the spring constant is not varied by strain, the C/C spring coil after 150 Mrad-EB irradiation is applicable for precise mass balance.

Fig. 6 shows change in spring constant at 0.15 of strain against irradiation dose. The EB irradiation enhanced the spring constant below 150 Mrad. Excess EB irradiation above ISO Mrad slightly decreased the spring constant.

4. Discussion

4.1. Stress relaxation of graphite structure

When an untreated C/C composite coil, having a graphite hexagonal structure with dangling bonds, was tested for tensile strength, a low spring constant was found in the range below 0.15 of compressive strain, as shown in Fig. 4. It was probably caused by the repulsive force of inter-sigma bonding electrons in a carbon atom with a dangling bond in a graphite hexagonal structure, as shown in Fig. 7. In particular, the repulsive force between sigma bonding electrons at a graphite carbon atom with a dangling bond should generate the stress



Figure 7 Schematic atom model before and after 150 Mrad-irradiation of electron beam. Stress relaxation of graphite is generated by the repulsive force of inter-sigma bonding electrons of the carbon atom with dangling bond.

relaxation. Thus, the low elastic stress with large strain observed in the stress-strain curve of an untreated C/C composite coil (see Fig. 4) could be explained.

4.2. High resistance to elastic deformation induced by EB irradiation

As shown in Fig. 4, the high resistance to compressive stress was found for the C/C composite coil spring treated at 112 Mrad-irradiation. High elasticity and straight stress-strain curve were also obtained. In order to explain the influences of EB irradiation on spring constants, the concept of annihilation of vacant sites in graphite hexagonal structure was suggested. If the EB treatment forcibly diffuses carbon atoms into vacant sites and incoherent interface because of high electrical potential (170 kV), such carbon atom migration probably decreased the vacant sites with dangling bonds. Namely, the EB irradiation decreased the dangling bond density in carbon fiber [5]. These observations were explained by annihilation of dangling bonds. In this case, the annihilation brought about high elasticity in carbon fibers irradiated by the electron beam, because the stress relaxation generated by the repulsive force of inter-sigma bonding electrons of the carbon atom with dangling bond didn't act in the treated carbon materials (see Fig. 7). Since carbon atoms with dangling bonds have stress relaxation induced by the enlargement of bonding angles, stress relaxation would not be expected in the EB irradiated C/C composite coil spring, which have a low density of dangling bonds. Thus, a high resistance to elastic deformation should be obtained for the EB irradiated C/C composite coil spring.

To simplify the discussion, we suggested that the effect of EB irradiation was mainly induced by migration of terminal carbon atoms in this paper. However, carbon fibers were generally contaminated by small amounts of nitrogen, hydrogen, oxygen, hydroxide and carbonhydride molecules, although heat treatment to reinforce the C/C composite removed most of the impurity atoms.

Fig. 8 shows ESCA peaks of graphite carbon atoms and carbon hydro-oxides for carbon fiber before and after EB-irradiation. Irradiated carbon fibers were also contaminated by small amounts of oxygen, carbonoxide and carbon-hydro-oxide molecules. Since the C–O bonding force is stronger than that of C–C



Figure 8 ESCA peaks of graphite carbon atoms and carbon hydrooxides for carbon fiber before and after EB-irradiation.

bonding, the impurity atoms were often trapped by vacant site with dangling bonds. If the oxygen atom occupied the vacant sit instead of carbon atom in graphite structure, the high spring constant induced by EB irradiation was also explained.

5. Conclusions

To enhance the spring constant, the electron beam irradiation was homogeneously performed. The EB irradiation of 150 Mrad largely enhanced spring constants, which were about two times higher than that before EB irradiation. The linear relationship was also obtained between compressive stress and strain. The excess EB irradiation above 150 Mrad slightly decreased spring constant of C/C composite coil springs irradiated at different EB irradiation doses.

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References

- 1. J. D. BROOKS and G. H. TAYLOR, Carbon $\mathbf{3}$ (1965) 185.
- 2. T. CHANG and A. OKURA, Transactions ISIJ 27 (1965) 229.
- 3. C. R. ROWE and D. L. LOWE, *Bienn. Conf. Carbon* **13** (1977) 170.
- 4. M. H. COHEN and D. TURNBULL, J. Chem. Physics. 43 (1965) 139.
- 5. Y. NISHI, T. TORIYAMA, K. OGURI, A. TONEGAWA and K. TAKAYAMA, J. Mater. Res. 16 (2001) 1632.
- 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. Mater. Res.* **13** (1998) 3368.

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