Influence of Environmental Temperature on Fatigue Reliability at Elevated Temperature for Carbon-Fiber-Reinforced PEEK*

(On the Growth Mechanism from Mesocracks to Macrocracks)

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In this paper, the influence of environmental temperature on fatigue reliability for advanced carbon-fiber-reinforced-thermal-plastic PEEK was examined. Attention was focused on the mechanism of growth from mesocracks to macrocracks at elevated temperature. The main results were as follows, (1) The fatigue life at elevated temperature was arranged by the fatigue strength of CF15 and gf30 materials decreased with increasing environmental temperature. Further, the strength of CF15 material was superior to that of gf30 material. (2) The influence of stress and environmental temperature was represented by the $N_f - 1/T$ diagram and $S - T(C + \log t_r)$ diagram. Therefore, it was suggested that high-cycle fatigue life could be estimated from these parameters. (3) From the results of fractography using SEM, the fracture surface of CF15 material was ductile, on the other hand, that of gf30 material was brittle. (4) It was appeared that the temperature rise in the fatigue damage zone was closely related to the mesocrack formation process. (5) It was thought that the difference of mesocrack formation between CF15 material and gf30 material was reflected in the difference of the activation energy.

Key Words: Fatigue, Reliability, Reinforced Plastics, Fatigue at Elevated Temperature, Fractography

1. Introduction

Recently, advanced fiber-reinforced-thermal-plastic (F RTP) having a high temperature tolerance and better damage resistance has been developed, and then, it has been discussed the fatigue reliability⁽¹⁾⁻⁽¹⁴⁾. Authors has conducted studies about fatigue reliability of these F RTP in systematically⁽¹⁾⁻⁽⁶⁾. In the previous studies⁽⁷⁾⁻⁽⁹⁾, a fatigue reliability of carbon fiber (CF) or glass fiber (gf)-reinforced nylon 46 (N46) has been tested on loading condition at high environmental temperature. N46 is advanced compos-

ite material having medium properties between engineering-plastics and super-engineering-plastics. Those materials have been estimated in experimentally and in systematically, especially a temperature rising⁽¹⁵⁾⁻⁽¹⁹⁾ of specimen with relation to its a fatigue damage process.

Then, the following results were obtained exactly about the fatigue reliability of N46 at the smooth notch specimen. CFR-N46 materials were superior to that of gfR-N46 materials. N46 materials having a high temperature tolerance was superior to that of N66 materials. The fatigue reliability of these materials have a dependence on environmental temperature. Most of fatigue life at high temperature was occupied with stage of mesocrack formation. On the other hand, at the shape notch specimen, most of fatigue life was the process of macrocrack propagation, so the fatigue life of these materials were nearly equal small.

Through these study, it was pointed out that the

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analysis of fatigue damage mechanism from mesocracks to macrocracks was significant subject in relation to the improvement and the estimation of fatigue reliability at elevated temperature on FRTP.

In recently, super engineering plastics having high temperature tolerance have been more expected to use to the high temperature parts and high strength parts in the engine room of automobiles. But, the fatigue reliability at high temperature for these plastics have not been cleared. Therefore, in this paper, the fatigue reliability for carbon-fiber-reinforced PEEK that is one of the super-engineering-plastics were examined at various environmental temperature. Moreover, this study was examined on elucidation to the mechanism of fatigue. In particular, the process of fatigue damage was analyzed at real-time. This test was persuaded by system that was combined with infrared thermography and fatigue test at elevated temperature by personal computer. Then this study was focused on the mechanism of growth from mesocracks to macrocracks at elevated temperature.

2. Experimental Procedures

2.1 Materials and specimens

The materials used in this investigation was a PEEK (poly (ether ether keton)). This materials is reinforced with carbon fiber of 15 weight% (CF15 material) or glass fiber of 30 weight% (gf30 material). Carbon fiber diameter was $7\text{ }\mu\text{m}$, initial length was 10 mm. Glass fiber diameter was $10\text{ }\mu\text{m}$, initial length was 3 mm. These materials were made into shape of dumbbell test specimen as shown in Fig. 1. The specimens were made of injection molding plate. Fiber orientation of injection molding plate on the surface layer (skin layer) was parallel to the injection direction. While the fiber orientation of the center layer (core layer) was perpendicular to injection direction. The stress concentration factor α was about 1.66. The main thermal properties of matrix were as shown in Table 1.

2.2 Fatigue test

Fatigue tests at high temperature were carried

Fig. 1 Shape and dimension of specimen

out using the fatigue testing system which was systematized by a servohydraulic testing machine, a high controlled temperature vessel and a infrared radiometer as shown in Fig. 2, with a $P_{\min}/P_{\max}$ load ratio R of 0.1 at 10 Hz were performed under ambient conditions ($T=373\text{ K}$, 393 K , and 413 K , is below glass transition temperature (T_g) of PEEK). This testing system was able to pursue a fatigue damage process on the surface of specimen at notch bottom at real time. The temperature rising of the surface of specimen at notch bottom has been measured with using infrared radiometer with accuracy of 0.05 K . Further the fracture surface after rupture of specimens were examined by using scanning electron microscopy (SEM).

3. Experimental Results

Figure 3 shows a experimental results of fatigue test of a relation between stress amplitude (S) and the number of cycles to failure (N_f) for PEEK composite (CF15 and gf30 material) at 373 K , 393 K and 413 K . The relation between S and N_f can be expressed exponential series of b by Eq.(1). The a is the material constant, and superscript b is the slope of strait line. The value of a and b are shown in Table 2.

$$S = aN_f^{-b} \quad (1)$$

The fatigue strength of both CF15 and gf30 materials decreases with an increase in environmental temperature. The decreasing rate of fatigue strength of CF15 and gf30 materials are 11% and 16% at 393 K ,

Table 1 Thermal properties of matrix

Matrix	PEEK
Glass transition temperature (K)	413
Thermal deformation temperature (K)	410
Melting point (K)	607

Fig. 2 Fatigue testing system

Table 2 $S=aN-b$

	CF15			gf30		
	a	b	S(Mpa) N=10 ⁷	a	b	S(Mpa) N=10 ⁷
373K	17.5	0.43	24.4	15.4	0.49	17.1
393K	15.8	0.4	22	12.9	0.41	14.4
413K	15.8	0.49	18	13.8	0.49	13.9

Fig. 3 $S-N$ curves

and are 26% and 19% at 413 K in comparison with the fatigue strength at 373 K. Moreover CF15 materials are superior to gf30 materials at each temperatures. That is, CF15 materials are superior in fatigue strength of 43% at 373 K, of 53% at 393 K and of 30% at 413 K to gf30 materials. The fatigue strength of CF15 at 413 K is larger than that of gf30 at 373 K. Therefore, it was understood that the more the temperature is high, the more the fatigue life is short, and carbon fiber is superior to glass fiber on effect of reinforcement.

According to these results, we try to make it clearly that the fatigue reliability at high temperature of PEEK composites would be considered from the point of view of mesoscopic and macroscopic.

4. Discussion

4.1 Influence of stress and environmental temperature on fatigue strength at elevated temperature

To explain a fatigue reliability of PEEK composites from the point of view of macroscopic, the results of $S-N$ curve was arranged by the Arrhenius model. The fatigue life (Nf) versus the environmental temperature (T) was plotted in Fig. 4. Stress amplitude was used as a parameter. From this figure, the relation between Nf and T could be expressed by Eq. (2).

$$\ln Nf = \alpha + \beta/T \quad (2)$$

The α is the material constant, and β is the slope of

Table 3 $\ln N = \alpha + \beta/T$

(Mpa)	CF15		gf30	
	α	$\beta \times 10^4$	α	$\beta \times 10^4$
S=60	-52.0	2.48	-	-
S=50	-41.6	2.23	-27.7	1.43
S=40	-	-	-33.0	1.81

Fig. 4 $Nf - 1/T$ diagram

straight line and correspond with the activation energy. The values of α and β are shown in Table 3. From Fig. 4, the curves for CF15 and gf30 materials were straight. The fatigue life increased in proportion to the reciprocal of environmental temperature in the range 373 to 413 K. So, it was thought that fatigue fracture mechanism of each materials were influenced of environmental temperature in this range. Besides, the slope of CF15 materials were larger than that of gf30 materials. It showed that the activation energy was influenced of the difference of fatigue strength.

Then, with considering the influence of creep, the relation of various stress, environmental temperature and fatigue life was arranged by the relation stress amplitude, S vs. Larson-Miller-parameter; $T(C + \log t_r)$ as shown in Fig. 5. $T[K]$ is absolute temperature, and $t_r[h]$ is the rupture time (it is changed [number of cycles to rupture/frequency] to time). From Fig. 5, the relation between S and $T(C + \log t_r)$ can be expressed by Eq. (3).

$$S = p - q \cdot T(C + \log t_r) \quad (3)$$

The p and C ($C=20$) are the material constant, and q is the slope of straight line. The values of p and q are shown in Table 4. It was thought that the influence of environmental temperature on fatigue strength at high temperature under T_g of tested PEEK composites could be arranged by these parameter, and long fatigue life could be estimated by these results.

Then, in the next paragraph, the relation between temperature rise on surface of specimens and fatigue damage were discussed to clear the temperature

Table 4 $S = p - q \cdot T(C + \log t_r)$

CF15		gf30	
p	$q \times 10^3$	p	$q \times 10^3$
82.5	7.15	68.5	6.08

Fig. 5 $S - T(C + \log t_r)$

Fig. 6 Photograph using SEM of fracture surface

dependence of stress and fatigue life were shown physically.

Fig. 7 Temperature rising curves of PEEK

4.2 Fractography with using SEM

The fracture surface after rupture of specimens were examined by using SEM. In particular, this observation was focused on the explanation of effects of mesoscopic factors which were fiber and matrix on a fatigue reliability at elevated temperature. The fatigue fracture surfaces of CF15 materials were shown in Fig. 6 (a), and that of gf30 materials were shown in Fig. 6 (b). These were examined at 373 K and 413 K.

The fracture surfaces of CF15 materials in Fig. 6 (a) showed that matrix resin was ductile and elongated remarkably. And it was recognized that the carbon fibers were pulled out from matrix and the fracture surface had many hole which the carbon fibers had been pulled out. That is, the fracture surface of CF15 materials were uneven. On the other hand, the fracture surfaces of gf30 materials in Fig. 6 (b) showed that matrix resin was brittle and was not elongated, and many glass fibers were ruptured. So, the fracture surface of gf materials were flat.

Further, on the both materials, the more at elevated temperature became high, the more the fracture surface were ductile. It was recognized that the fracture surfaces were influenced of kinds of fibers and environmental temperature. Therefore, it was understood that the fatigue life of both materials were influenced of differences of these fracture surfaces.

4.3 Chase of the fatigue damage process by temperature rise

The temperature rising of the surface of specimen at notch bottom has been measured with using infra-red radiometer through the fatigue test. The temperature rising $\Delta T (= T_m - T_r)$ were plotted against number of cycles N as shown in Fig. 7. T_m was maximum temperature of specimen surface, and T_r was environmental temperature. It was recognized that

the temperature rising changed with increase in stress cycles as follows, the first (I) stage; the temperature showed rising rapidly by initial cyclic deformation. The third (III) stage; the temperature showed rising rapidly too and the specimen was ruptured finally.

Next, the second (II) stage; the variation of temperature was nearly equal constant. And this stage occupied most of fatigue life. The life of the II stage on CF15 materials were about from 80% to 90%, and that on gf30 materials were about from 60% to 70%. Therefore the II stage is the most important on fatigue reliability of these specimens. Further, the temperature rise were decreased on the II stage at the all environment temperature of both CF15 materials and gf30 materials. The CF15 material at 413 K was shown in Fig. 7 that was the most decrease of temperature, and this was 4K. The others were the decrease of 1 K.

Further, for the purpose of elucidation of these phenomena, the fatigue damage zone of surface near the notch bottom of specimen on the II stage were observed in the range of 1 pixel ($180 \times 180 \mu\text{m}$) of a

thermal picture of infrared radiometer by using SEM. The SEM photograph and its sketch of surface of specimen were in Fig. 8. This figure shows the results of CF15 materials at (a) 373 K, (b) 393 K, (c) 413 K and (d) no-load. It was recognized that the mesocracks was initiate on the surface, and most of it were initiated from the interface between fiber and matrix, the end of fiber, and the part of crossing fibers. The quantity of mesocracks increase with rising temperature, and the length of cracks increase too at 413 K. And gf30 materials showed same tendency for CF15 materials, but the number of mesocracks was fewer than CF15 materials. So it was thought that the cause of temperature decreasing on the II stage was to become easy to diffuse a heat due to increasing of surface area following the initiation and the increasing of mesocracks.

Therefore it was thought that the temperature rise form was closely related the mesocracks formation in the II stage which occupied the most of fatigue life. Then, the schematic illustration of relation between temperature rise and fatigue damage was observed as follows and as shown in Fig. 9. The first, the temperature rise was decreased a little when initial mesocracks was initiated (①). Next, the temperature rise was decreased as the mesocracks increased (②, ③). Finally, a macrocrack initiated and propagate with mesocracks coalescence (④). Hence, the transition of temperature rise of fatigue life relate to the fatigue damage accompanying the mesocracks

Fig. 8 Photograph using SEM of surface of test piece of CF15

Fig. 9 Schematic illustration of fatigue damage on the II stage

Fig. 10 Schematic illustration of relation between activation energy and the growth from mesocrack to macrocrack

initiation and growth. It was suggested that these transition of temperature were influenced by the creep deformation.

4.4 Relation between activation energy and fatigue damage

In previous paragraph, it was recognized that fracture mechanism on the II stage was not influenced of environmental temperature, and all specimen has almost same transition of fracture which followed the mesocracks initiation, but the number of mesocracks of gf30 materials were fewer than CF30 materials. Additionally, the II stage held about 80–90% of fatigue life on CF15 materials. On the other hand, it held about 60–70% of fatigue life on gf30 materials which were shorter than CF15 materials at each temperature.

Therefore, it was suggested that a power of resistance against initiation of mesocracks of CF15 material was bigger than that of gf30 material, and the difference of fatigue life between CF15 material and gf30 material was influenced of this resistance. Figure 10 is schematic illustration of relation between fatigue damage and activation energy. This difference of resistance between CF15 and gf30 material was equivalent to the difference of activation energy of between CF15 and gf30 material shown in Fig. 4. That is, the activation energy of CF15 material was more than that of gf30 material. From this figure, CF15 material which has many amount of mesocracks initiation had large activation energy what was called contributing energy to fatigue damage. On the other hand, gf30 material had little amount of it had small activation energy. Hence, the difference of fatigue life between CF15 and gf30 material is explained by difference of activation energy.

5. Conclusion

In this paper, the influence of environmental temperature on fatigue reliability for carbon-fiber-rein-

forced PEEK was examined. Attention was focused on the mechanism of growth from mesocracks to macrocracks at elevated temperature. The main results were as follows, (1) The fatigue life at elevated temperature was arranged by following equation: $S = aN_f^{-b}$. The fatigue strength of CF15 and gf30 materials decreased with increasing environmental temperature. Further, the strength of CF15 material was superior to gf30 material. (2) The influences of stress and environmental temperature were represented by the $N_f - 1/T$ diagram and $S - T(C + \log t_r)$ diagram. Therefore, it was suggested that high-cycle fatigue life could be estimated from these parameters. (3) From the results of fractography using SEM, the fracture surface of CF15 material was ductile, on the other hand, that of gf30 material was brittle. (4) It was appeared that the temperature rise in the fatigue damage zone was closely related to the mesocrack formation process. (5) It was thought that the difference of mesocrack formation between CF15 material and gf30 material was reflected in the difference of the activation energy.

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