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Carbon / Epoxy 적층판 T300/914($\pm 45°$)$_{2S}$,의 Fatigue와 Fatigue-Creep에서의 Acoustic Emission에 대한 Matrix의 역할

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Role of the Matrix for Acoustic Emission in Carbon / Epoxy Laminates T300 / 914($\pm 45°$)$_{2S}$, in Fatigue and Fatigue-Creep

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요 약 : Fatigue와 fatigue-creep 하에서 T300 / 914 ($\pm 45°$)$_{2S}$의 acoustic emission (A. E.)과 damage development가 연구되었다. Ductile한 914매트릭스가, 매트릭스 특성의 A. E.와 damage development에 대한 영향을 살펴보기 위하여 선정되었는데, 이 매트릭스 시스템의 ductility는 매트릭스 내에 산재한 열가소성 nodule에 의한 것이다. Fatigue에 있어서의 damage development는 서로 다른 네 단계를 거쳐서 최종파괴에 이르고, fatigue-creep에 있어서는 세 단계를 거친다. 이러한 차이점은 creep interruption에 의한 stress-relaxation과 stress-redistribution에 의한 것이다. 이는 fatigue-creep에 있어서 fatigue-life를 증대시키고 A. E.를 많이 감소시킨다.

Abstract : The acoustic emission (A. E.) and the damage development in T300/ 914, ($\pm 45°$)$_{2S}$ were investigated in fatigue and fatigue-creep loading. The ductile matrix system of 914, in which this ductility comes from the thermoplastic nodules dispersed in the matrix, is chosen to investigate the inherent matrix properties for A. E. and damage development. The damage development in fatigue passes through four different steps to the final rupture. It goes through three different steps in fatigue-creep. This difference comes from the stress relaxation and stress redistribution by the creep interruption. This provides a prolonged fatigue life and much less active A. E. in fatigue-creep.

INTRODUCTION

When a composite material is subjected to external loading, there is a characteristic release of energy, primarily in the form of acoustic, thermal and exo-electronic emissions.[1,2] By attaching a piezoelectric transducer to the specimens, these acoustic signals can be detected, electronically processed, and then recorded throughout a given test. The understanding for the origin of these acoustic signals has been much improved since 1964.[3] At this time, the connection between amplitude distribution and source mechanism is not very well known.

The typical damage mechanisms in fibrous composites are rupture of fiber-matrix interface, matrix cracking, rupture of fibers, and the delamination. The magnitude of A. E. signals emitted by fiber breakage is generally greater than that of the others.[2] This fact could be an obstacle to the proper analysis of matrix cracking and rupture of fiber-matrix interface.[4-7] For these cases the role of matrix is very important. And the A. E. results in complex loading systems are not sufficient.

The composite materials T300/914 with the structure of $(\pm 45^\circ)_{2S}$ were tested in the complex loading systems, fatigue and fatigue-creep. The matrix of 914 is known very sensitive to the creep and the plastic deformation by its nature.

EXPERIMENTAL METHODS

Materials

T300/914 with the stacking sequence of $(\pm 45^\circ)_{2S}$, $+45^\circ/-45^\circ/+45^\circ/-45^\circ/-45^\circ/+45^\circ/-45^\circ/+45^\circ$ was supplied by ETCA of France. The fiber volume fraction is 0.63. Straight sided coupon specimens with 235mm long and 25mm wide were prepared and its gage length is 135mm. The side edges of the specimens were ground with sand papers and polishing powders to eliminate the surface flaws. The glass/epoxy tabs were used. These specimens were stored in a conditioning cabinet with relative humidity (R.H.) 65% at 23℃ before use.

Acoustic Emission Detection

The A. E. system is a Dunegan 3000 series detector to which signals from the piezo-electric transducer are fed after pre-amplification through a Dunegan S/D-60 p pre-amplifier. The transducer is a broadband pick-up(0-300 kHz) with a resonant frequency of 120kHz. The transducer is held firmly against the specimens by a gripping device with silicon grease as a coupling medium. The transducer signal is pre-amplified 40 dB and filtered below 100kHz. Total amplification is 70dB with threshold of 0.5 V. The glass/epoxy end tab was chosen to avoid noisy shear debonding in the grips. The scheme is shown in Fig. 1.

Mechanical Testing

Mechanical testing was carried out in a floor-model Instron. The fatigue loading ($f=2$Hz, triangular form, $R=0.1$) was applied with the constant maximum

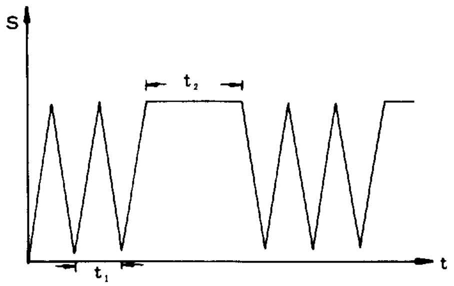


Fig. 1. Fatigue-creep loading system. ($t_1=0.5$ sec,, $t_2=150$ sec)

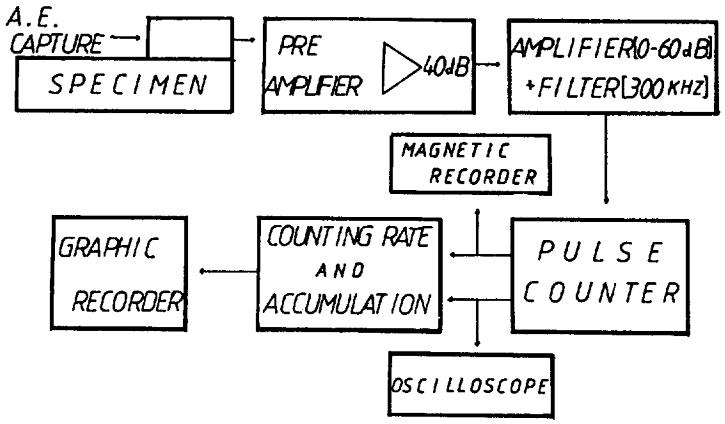


Fig. 2. Scheme of acoustic emission apparatus.

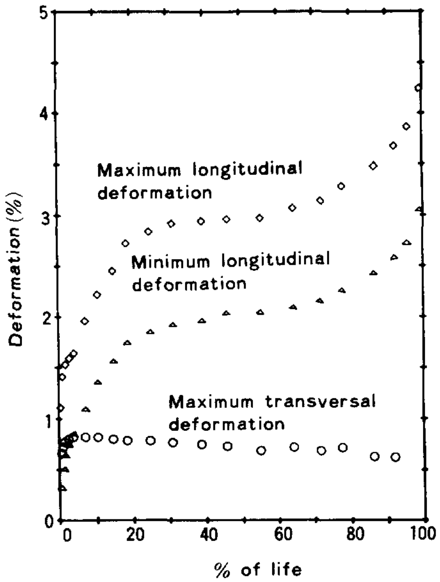


Fig. 3. Variation of deformation in fatigue.

Table 1. Composition of 914

Constituent	Nature*	Parts
Epoxy	TGMDA(MY-720)	40
Epoxy	TGPAP	60
Hardener	DICY	5
Additive	PES	40

*TGMDA : Tetraglycidyl methylene dianiline
TGPAP : Triglycidyl P-aminophenol
DICY : Dicyandiamide
PES : Polyethersulfone

stress, 125 $\mathrm{MP_a}$ · The strain gages were used to detect the deformations of the specimens.

For fatigue-creep loading, the fatigue cycles were regularly interrupted by the creep loading (see Fig. 2). Computer control technique for Instorn was used to generate this loading type. The fatigue loading was interrupted every 300 cycles. The creep period were always 150 seconds. Thus the fatigue and creep period were equally maintained in this study.

RESULTS AND DISCUSSION

The composite materials T300 / 914 were chosen by its particular matrix system. There are dispersed thermoplastic nodules in this matrix. It's composition is represented in Table 1.[8]

And it's characteristics are very sensitive to the curing conditions and the environmental exposure.[8] To ensure the consistency of data all of the

In this study the maximum stress is 125MPa. This load level was chosen by the results of monotonic tensile test and A. E. .[9] The damage growth is not very fast for this load level. The variations of deformations in fatigue are showm in Fig. 3.

The longitudinal deformation increases very quickly until the 17% of life. And then it slows down to the 70% of life. It's variation is very small. After this period it starts to increase. The transversal deformation increase slightly until 4% of life. At this point it begins to decrease for the final rupture. These results suggest that there may be 4 different steps in the damage development.

To prove above phenomena the A. E. are recorded until the final rupture by using 4 specimens. Their fatigue lives are 9860, 13240, 13737 and 14578 cycles. It's nearly constant except one specimen. But their trend for A. E. as a function of life is nearly the same. These results are demonstrated in Fig. 4. The cumulative number of counts is related to the extent of damage. And the rate

specimens used in this study are cut from the same panel.

of counts is related to the rate of damage growth. So we have chosen the rate of counts to explain the damage growth mechanisms.

From the beginning to the 3% of life, there are several A. E. It would come from the damage occurrence in the fiber-matrix interface.[10-12] The following short stops of A.E.show the stress relaxation effect. The A. E. becomes very intensive from 7% to 18% of life. In this nodular system the shear strength of matrix-nodule interface is lower than that of fiber-matrix interface. The first appearance of a few damage site in the fiber-matrix interface can be attributed to the inherent characteristics of this composite. The sites of matrix-nodular separation are numerous. This fact provides an intensive A. E. After this period the A. E. became weak and intermittent to 52% of life.

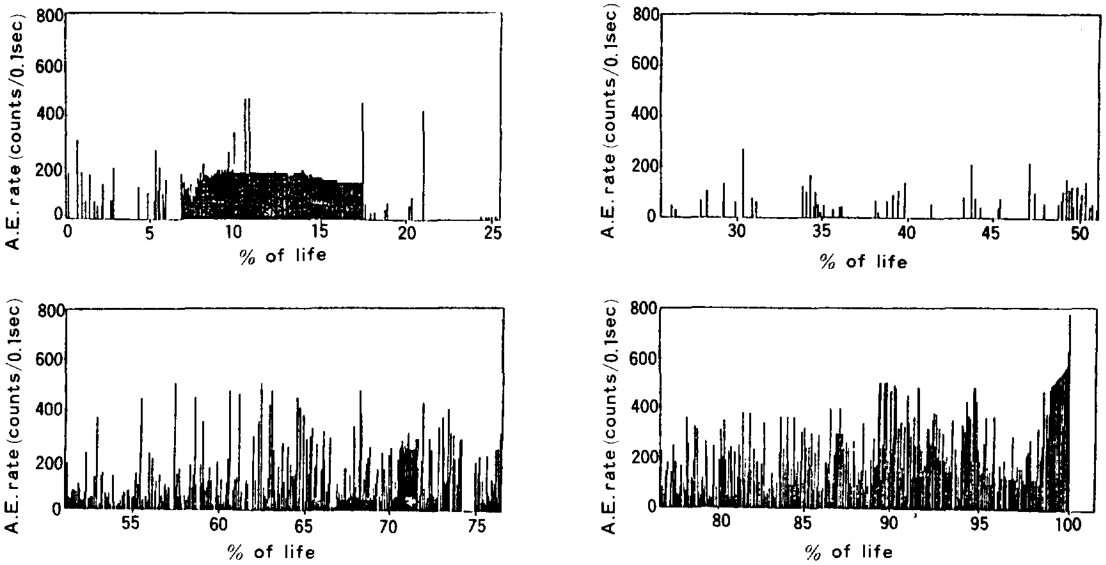


Fig. 4. Acoustic emission in fatigue test (f=2Hz, triangular, R=0.1).

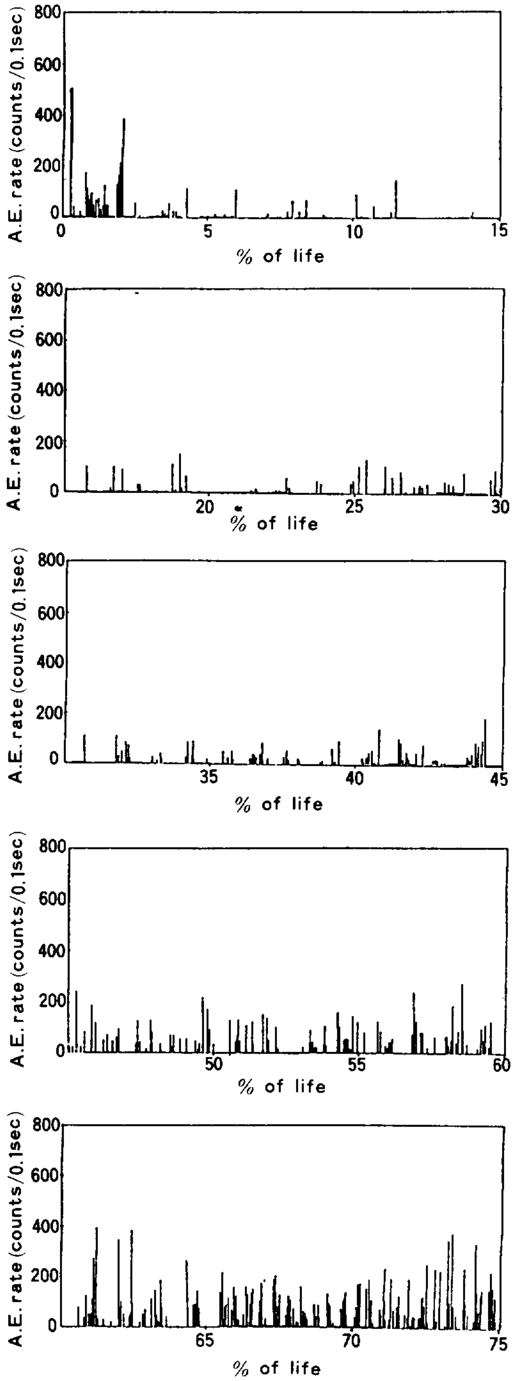
There may be two reasons. One is the great multiplication of the damage site and the other is the pronounced plastification in the crack tips. The damaged matrix can also attenuate the A. E. The pronounced plastification or ductility permits the fiber rotation. It gives an additional stiffness to the material. This results in the stagnation of the longitudinal deformation. The slight decrease of transversal deformation is attributed to the unstability of the specimen caused by the fiber rotation and by the matrix-nodular separation.

After this intermittent region the A. E. is active. In this period the damage in the matrix-nodular interface is saturated. The damage accumulation occurs in the fiber-matrix interface and in the form of intermittent fiber breakage.[9,10] The longitudinal deformation increases by this fiber breakage and by the increased damage of fiber-matrix interface. This fact shows that the A. E. is closely related to the micro-structural changes. It also shows the importance of the stress relaxation in the matrix for the A. E. To understand this effect the fatigue-creep tests have been performed for this matrix system.

We have chosen the loading history presented in Fig. 2 to get the largest fatigue-creep interaction. The previous work for A. E. with the creep loading interrupted with and without the relax period was done by Goyau.[13] The cumulative A. E. counts were represented such as the damage accumulation parameter. A. E. accumulation curves are common smooth parabolic curves. Only the slight frequency effect was shown. It was insufficient to get the detailed knowledge of the damage development. And the total cumlative A. E. counts for rupture vary with the frequency and the loading history.

In this study the A. E. counting rate was surveyed throughout the test. Three specimens from the same panel were used to detect the A. E. Two of these three specimens were tested until the final rupture. And another one is failed near the rupture by an accident. Their endurances are summarized in Table 2.

The above results show that the interruption of fatigue-creep may increase seriously the fatigue cycles to the rupture. It would come from the stress relaxation by the creep period and the stress redistribution by the fatigue-creep interaction.[9] The deformations were measured by strain gages. But with the large deformation and long endurance,



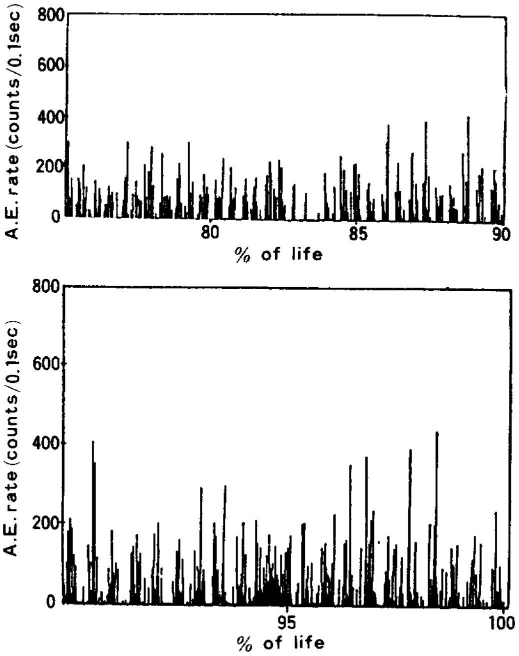


Fig. 5. Acoustic emission in fatigue-creep.

Table 2. Endurance in Fatigue-Creep

No ·	No. of fatigue cycles	Creep time (sec.)
1	44,400	22,050
2	39,000	19,350

the strain gages could not sustain until the final rupture. So these results are abbreviated.

The A. E. results in fatigue-creep are shown in Fig 5. The general mode of this A. E. is completely different from that of fatigue. This suggests that the damage development in these two loading systems is different. From the starting point to 2% of life the A. E. is active in the creep period. It means that the damage occurs in the fiber-matrix interface with the same reason in fatigue. But after this period until 66% of life the A. E. is very weak and intermittent. The stress relaxation by the creep and the stress redistribution by the fatigue are the main reasons for this fact. It would be also attributed to the resin flow near the fiber-matrix interface.[9] From the 66% of life to the final rupture the A. E. becomes somewhat more active. In this region the damage occurs both in the fiber-matrix interface and in the matrix-nodular inrerface. The damage occurs only in the creep period. It means that the fatigue loading does play a role of stress-redistribution. So the damage development in these creep periods has a tendency to be evenly distributed. This fact results in the prolonged fatigue life. The above discussions lead us to the conclusion that the damage development in fatigue-creep is different from that in fatigue. And the A.E. results should be correlated with the micro-structural changes in the materials.

CONCLUSION

The damage development in fatigue which was proven by the stress-strain curves and the A. E. passes through four different steps. In fatigue-creep it goes through three different steps in general. This difference comes from the stress relaxation and redistribution effect for the damage development. It also results in the prolonged life in fatigue-creep. The A. E. analysis should consider the nature of the material systems and the micro-structural changes.

REFERENCES

1. H. L. Dunegan and D. O. Harris, "AE Techniques' in "Experimental Techniques in Fracture Mechanics", ed. by A. S. Kobayashi (SESA 1973).
2. R. S. Williams and K. L. Reifsnider, *J. Compos. Mater.* **8**, 340 (1974)
3. B. Echalier, "Acoustic Emission and Modelisation of Damage. An Application for Glass / Epoxy Wounded Structure", in *JNC* 4, Paris, 1984.
4. J. Awerbuch, J. Block, and R. Prinz, "Effect of Stacking Sequence in Cross-Ply Graphite / Epoxy Laminates on Acoustic Emission Results", in *ICCM* 4, Tokyo, 1982.
5. A. Rotem, *Compos.* **33** (1978, Jan.).
6. R. D. Jamison, "On the interrelationship between Fiber Fracture and Ply Cracking in Graphite / Epoxy Laminates", in ASTM Symposium, Dallas, 1984.
7. D. Laroche and A. R. Bunsell, "Stress and time dependent damage in carbon fiber reinforced plastics", in *ICCM* **3**, Paris, 1980.
8. Y. S. O, Thesis for Doc. d'Etat INSA, 1984.
9. J. R. Lee, Thesis for Doc. Ing. U.T.C., 1985.
10. M. A. Zaid, Thesis for Doc. Ing. U.T.C., 1982.
11. F. A. Adams and D. R. Doner, *J. Comp. Mat.*, **1**, 4(1967).
12. R. L. Foye, *J. Comp. Mat.*, **7**, 189(1983).
13. G. Goyau and C. Cazeneuve, ETCA report 83-R-011.
14. C. T. Sun and E. S. Chim, *ASTM STP* **723**, 2333(1981).
15. M. Fuwa, B. Harris, and A. R. Bunsell, *J. Appl. Phys.*, **8**, 1460 (1975).
16. J. B. Sturgeon, *J. of Mater. Sci.*, **13**, 1490(1978).
17. P. Sigety and J. P. Favre, ONERA report 37. 7086M.
18. J. P. Favre, P. Sigety, and C. Marias, ONERA report N37, 285 / OM.
19. B. Testard, F. Ferry, P. Fleischmann, J. C. Rouais, and D. Rouby, "Analyses of acoustic emission signals produced in Carbon-Epoxy T300 / 5208 laminates", in *JNC* 4, Paris, 1984.
20. A. Rotem, *Compos. Tech. Rev.* **6**, 145(1984).
21. B. Ponsot and D. Valentin, "Acoustic Emission Monitoring of Damage in Unidrectional Carbon Resin Composites : Role of the Matrix", in *JNC* 4, Paris, 1984.
22. M. A. Hamstad, Report of Avracom, NTR 83-F-7.