Time and Temperature Dependence of Flexural Fatigue Strength for Pitch-based CFRP

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SUMMARY: The flexural fatigue behavior of three types of unidirectional CFRP laminates, combined with three types of pitch-based carbon fibers and single type of matrix epoxy resin, were evaluated at several levels of loading rate and temperature. The CSR (constant strain-rate) and the fatigue behavior of these CFRP were found to be remarkably dependent on time and temperature. The time-temperature superposition principle for the CSR strength of the CFRP holds also for these fatigue strengths, although these time-temperature shift factors do not always agree with that of the viscoelastic behavior of the matrix resin. The master curves of flexural fatigue strength for three types of CFRP were obtained, and the characteristics of these fatigue strengths were clarified from these master curves.

KEY WORDS: Pitch-based carbon fiber, CFRP, Flexural behavior, Fatigue, Time and temperature dependent properties

INTRODUCTION

It is well known that the mechanical behavior of polymer resins exhibits time and temperature dependence, called viscoelastic behavior, not only above the glass-transition temperature T_g but also below T_g. Thus, it can be presumed that the mechanical behavior of CFRP using polymer resins as matrices also significantly depends on time and temperature. It has been confirmed that the viscoelastic behavior of polymer resins as matrices is a major influence on the time and temperature dependence of the mechanical behavior of CFRP using a PAN-based carbon fiber [1-4].

Pitch-based carbon fibers have a high tensile modulus, because they are made from coal-tar pitch or petroleum pitch which have good graphitizability. Therefore, CFRPs using a pitch-based carbon fibers are being used as structural materials of aircraft, spacecraft, etc., where high modulus is required.
In this paper, the flexural fatigue behavior of three types of unidirectional CFRP laminates, combined with three types of pitch-based carbon fibers and single type of matrix epoxy resin, were evaluated at several levels of loading rate and temperature. The time and temperature dependence of the flexural fatigue behavior of these CFRP laminates is discussed from the viewpoints of the viscoelastic behavior of the matrix resin and characteristic mechanical behavior of pitch-based carbon fiber.

EXPERIMENTAL PROCEDURE

Preparation of Specimens

Unidirectional CFRP laminates using three types of pitch-based carbon fiber and two types of epoxy resin were prepared. The three types of pitch-based carbon fiber were Granoc[Æ] XN70, XN40, and XN05 (Nippon Graphite Fiber) as shown in Table 1. The matrix resins were general purpose epoxy resins 25C and 25P. The T_g of 25C and 25P is 140 and 132°C, respectively. The three kinds of prepreg sheets made from these fibers and resins, which are XN70/25C, XN40/25C, and XN05/25P (fiber/resin), were hot pressed into 3 mm thick unidirectional CFRP laminates. The fiber volume fraction of each CFRP laminates was 55%, 55%, and 48%, respectively.

Table 1: Specifications and mechanical properties of carbon fibers.

	XN70	XN40	XN05
Diameter of mono filament(μm)	10.0	10.0	10.0
Filament number in 1yarn	2000	2000	3000
Elastic modulus(GPa)	682	402	57.0
Tensile strength(GPa)	3.60	3.43	1.12
Ultimate strain(%)	0.50	0.85	1.80
Specific gravity	2.16	2.12	1.65

Test Procedures

Three point bending tests under constant strain-rate (CSR) and fatigue loadings for three CFRP laminates, XN70/25C, XN40/25C, and XN05/25P, were conducted at various loading rates and temperatures. The nominal dimensions of the test specimens are 80, 10, and 3 mm (length, width, and thickness). The span is 60 mm.

RESULTS AND DISCUSSION

Viscoelastic Behavior of Matrix Resin

Figure 1 shows the master curves of storage modulus E' versus reduced time t' at a reference temperature T_0=25°C, for two types of resin, 25C and 25P. These master curves were constructed by shifting the E' horizontally and vertically at various constant temperatures until they overlapped. Since E' at various temperatures can be superimposed so that a smooth curve is created, the modified time-temperature superposition principle is applicable for the E' of these resins.

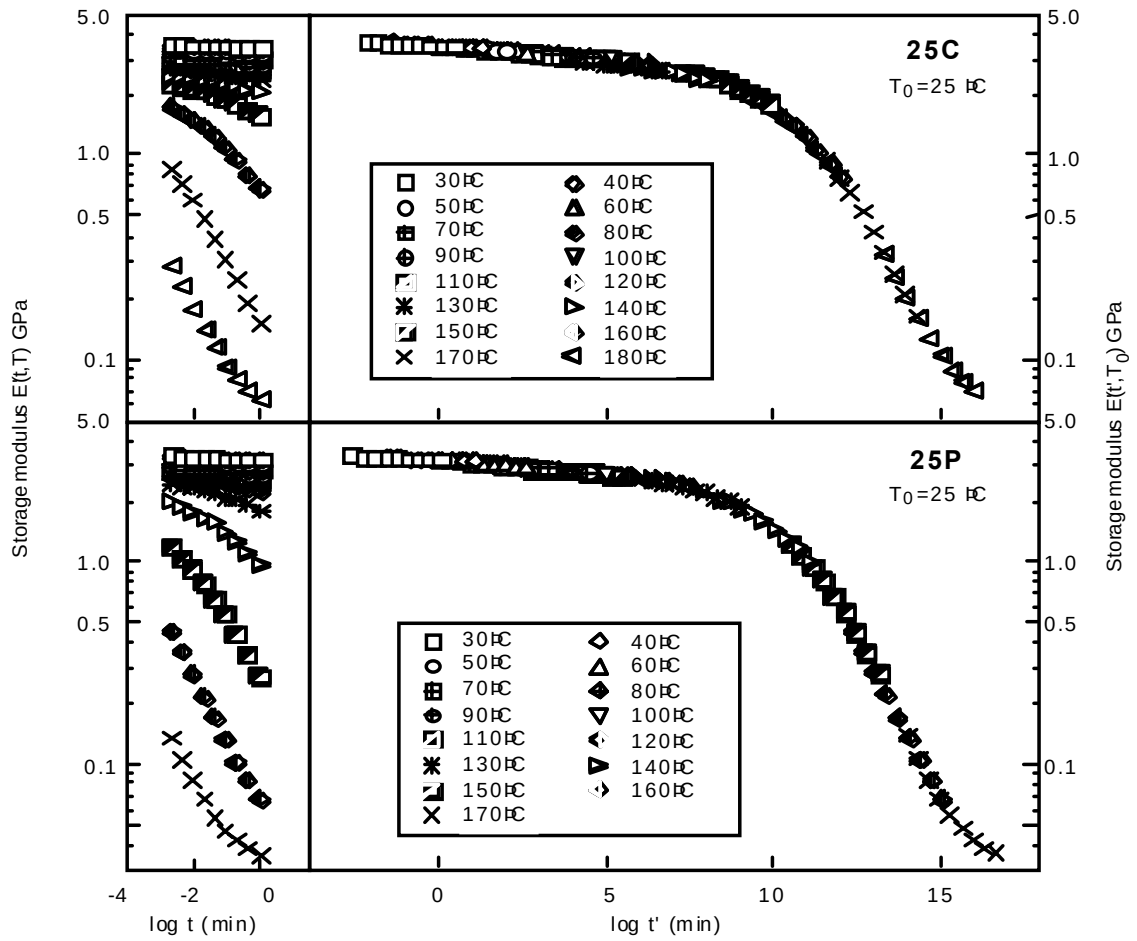

Fig.1: Master curves of storage modulus for matrix resin, 25C and 25P.

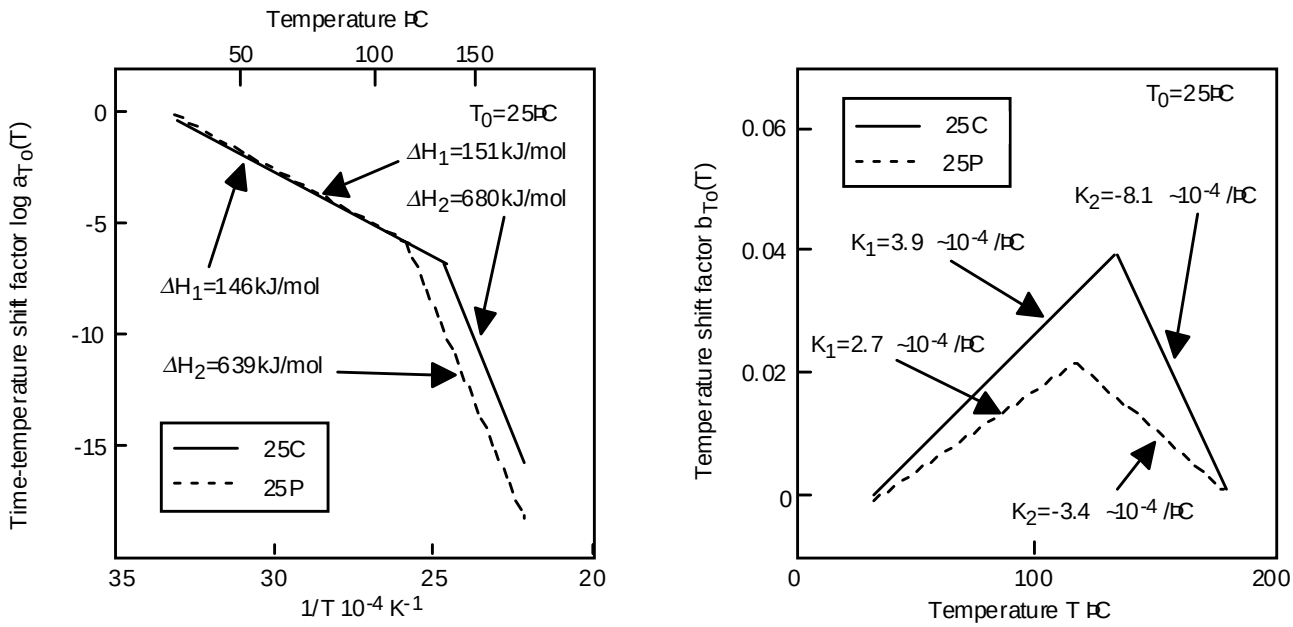

Fig.2: Shift factors $a_{To}(T)$ and $b_{To}(T)$ for storage modulus of matrix resin, 25C and 25P.

The horizontal time-temperature shift factor $a_{To}(T)$ and the vertical temperature shift factor $b_{To}(T)$ are shown in Fig.2. The $a_{To}(T)$ are quantitatively in good agreement with Arrhenius' equation by using two different activation energies ΔH. The temperature at the knee point of the two Arrhenius' equations corresponds closely to the T_g of each resin. The $b_{To}(T)$ can be described by two straight lines. The knee point temperature of the two straight lines also corresponds to the T_g of each resin.

Load-deflection Curves

Typical load-deflection curves for three types of CFRP laminate under CSR loading with deflection rate V=2mm/min are shown in Fig.3. The load-deflection curves for XN70/25C and XN40/25C using high modulus carbon fibers show non-linear behavior until maximum load. On the other hand, the load-deflection curves for XN05/25P using low modulus carbon fiber show linear behavior until maximum load. It is considered that this non-linear behavior on the load-deflection curve is caused by the remarkable non-linear behavior of high modulus carbon fiber in the compression side of specimen.

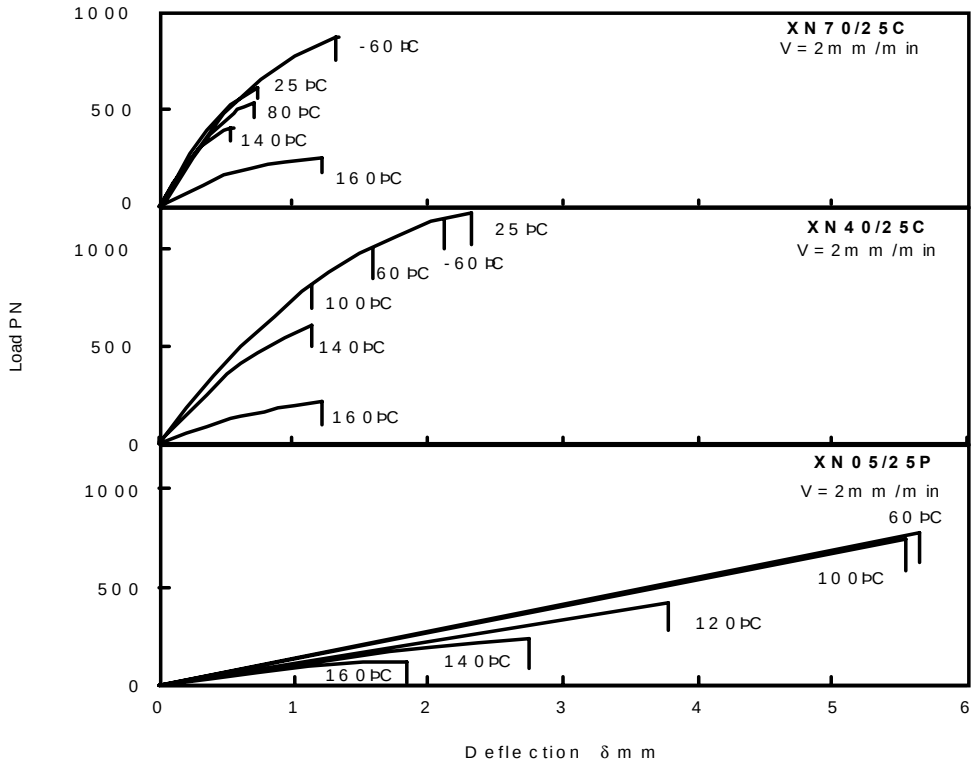

Fig.3: Load-deflection curves under the flexural CSR loading.

Flexural CSR Strength

The left side of Fig.4 shows the flexural CSR strength σ_s versus time to failure t_s at various temperatures T, and the right side shows the master curve of σ_s at T_0=25°C. From the left side graphs it is shown that the σ_s depends on t_s and T. The master curves of σ_s on the right side were constructed by shifting the σ_s at various constant T along the log scale of t_s they overlapped each other. The vertical shift applied in the case of the stress-strain relation of the matrix resin is not needed for the strength of the CFRP laminate because the applied load is

mostly transferred to the fiber of the CFRP laminate. Since the σ_s at various temperatures can be superimposed smoothly, the time-temperature superposition principle is also be applicable for the σ_s of three types of CFRP laminates.

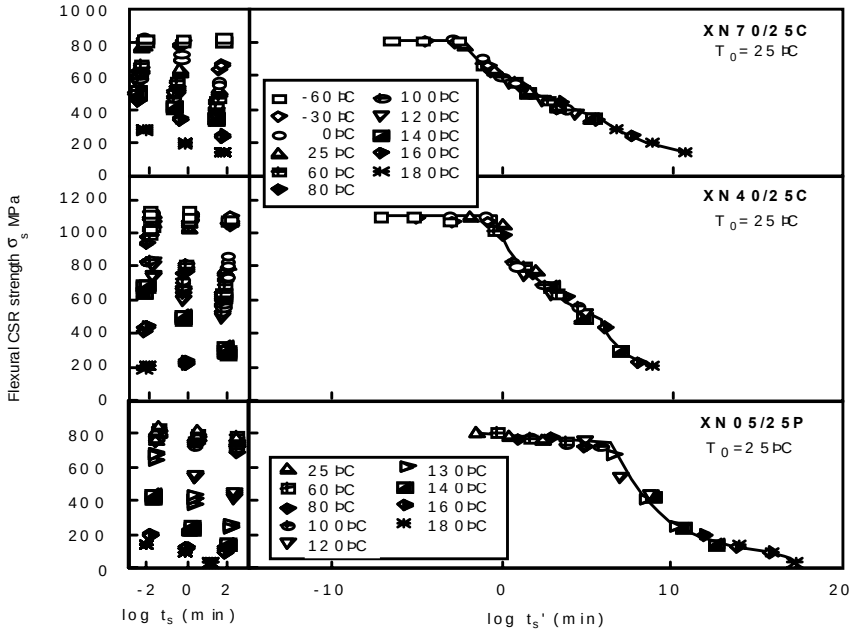

Fig.4: Master curves of flexural CSR strength for three types CFRP laminate.

Figure 5 shows the time-temperature shift factors $a_{To}(T)$ obtained experimentally for the master curves of σ_s. These shift factors are quantitatively in good agreement with Arrhenius' equation by using two different ΔH. The dotted lines in this figure show the $a_{To}(T)$ for the storage modulus E' of matrix resin in Fig.2. The $a_{To}(T)$ for the σ_s do not agree with that for the E' of matrix resin. It is presumed from this disagreement that the mechanical behavior of pitch-based carbon fiber itself shows time-dependent behavior.

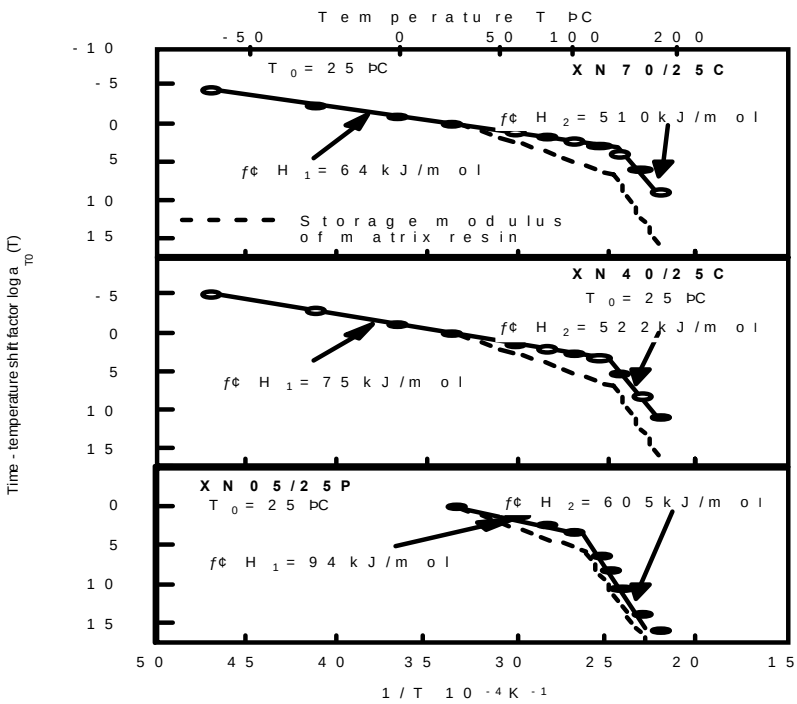

Fig.5: Time-temperature shift factors of CSR strength for three types CFRP laminates.

Figure 6 shows the classification of fracture mode on the master curves of σ_s. In the range of short t_s and low temperature, the tensile fracture occurs in the surface layer of the tension side of specimen for all CFRP laminates as shown in the left side of Fig.7,where the σ_s changes scarcely with t_s as well as temperature. In the range of intermediate time and temperature, the compressive fracture occurs in the surface layer of the compression side of specimen for XN70/25C and XN40/25C as shown in the center of Fig.7,where the σ_s changes remarkably with t_s as well as temperature. In the range of long t_s and high temperature, the microbuckling fracture of fiber occurs in the compression side of specimen for all CFRP laminates as shown in the right side of Fig.7,where the σ_s changes with t_s and temperature.

Flexural Fatigue Strength

Figure 8 shows the flexural fatigue strength σ_f versus the number of cycles to failure N_f (S-N curve) at a frequency f=5Hz for XN70/25C and XN40/25C, f=2Hz for XN05/25P. These S-N curves show also the characteristic curves in the ranges of N_f and temperature, each corresponding to a different mode of fracture. In the range of small N_f and low temperature, the tensile fracture occurs, where the σ_f changes scarcely with N_f as well as temperature. In

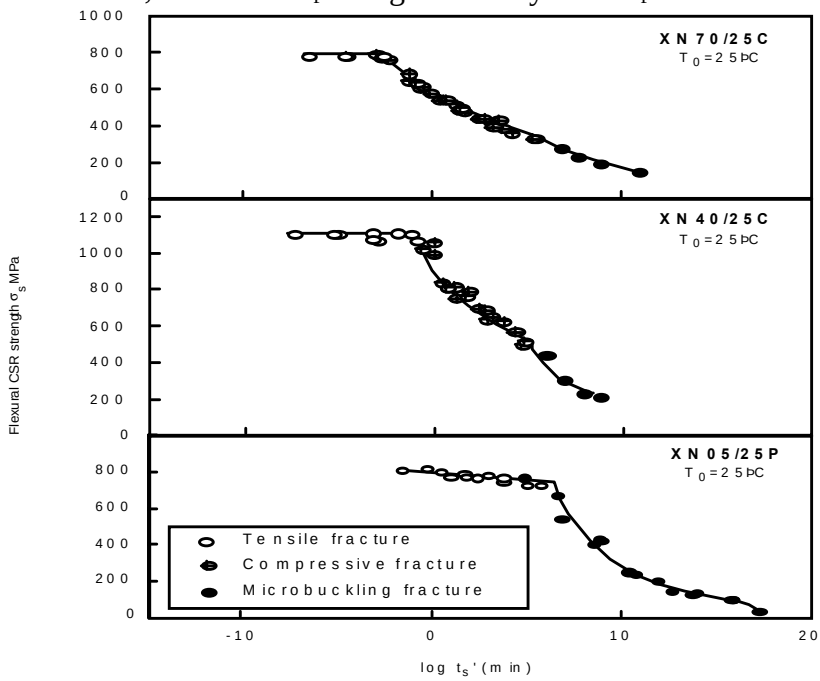

Fig.6: Master curves of flexural CSR strength for three types of CFRP laminate.

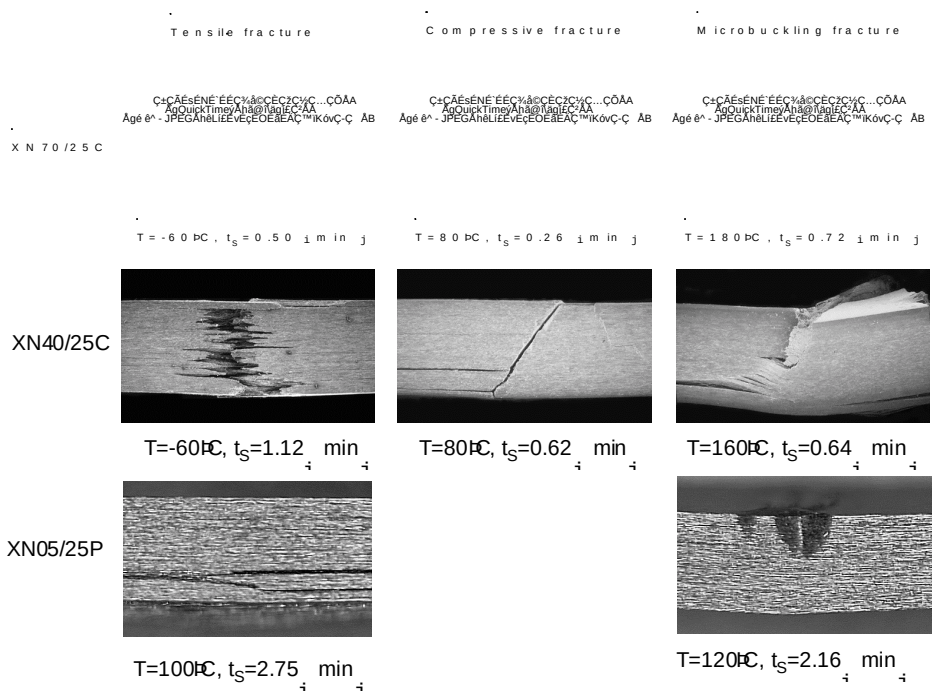

Fig.7: Fractographs after flexural CSR test for three types of CFRP laminate the range of large N_f or intermediate temperature, the compressive fracture occurs, where the σ_f changes remarkably with N_f as well as temperature. In the range of high temperature, the microbuckling fracture of fiber occurs, where the σ_f changes with N_f and temperature.

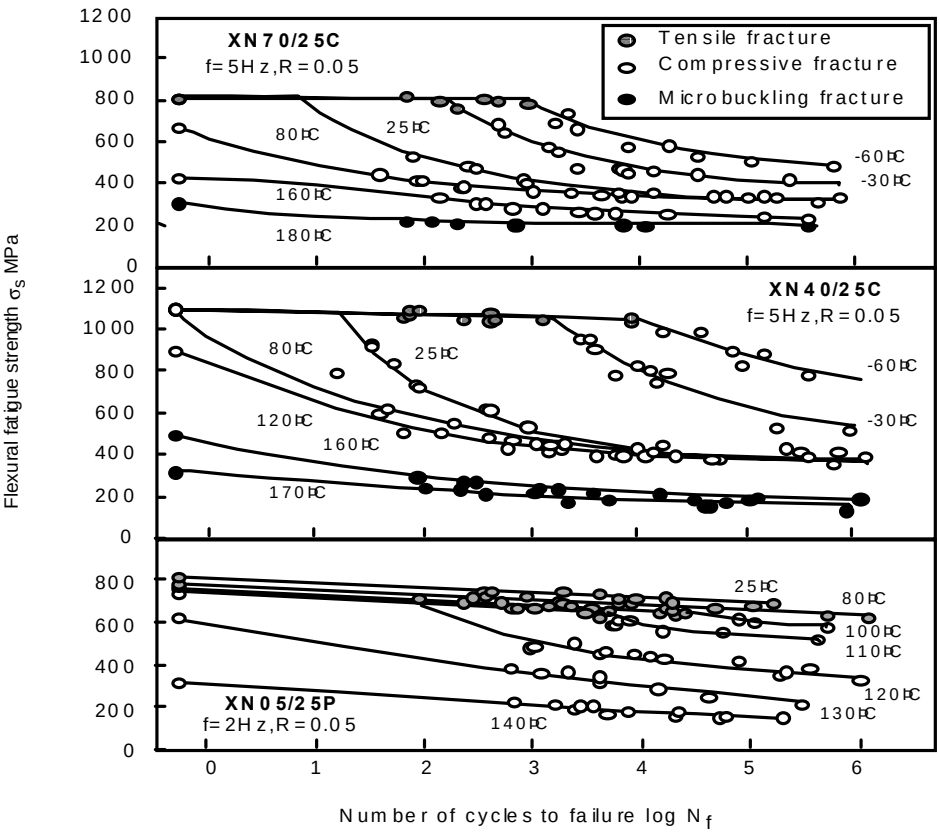

Fig.8: S-N curves of three types CFRP laminates at various temperatures.

The time-temperature superposition principle for the CSR strength σ_s of three types of CFRP laminates can be applied to the fatigue strength σ_f. Figure 9 shows the master curves of σ_f, which can be constructed by using the S-N curves in Fig.8 based on the time-temperature superposition principle for the σ_s. The master curves can be divided into three distinct groups of curves for XN70/25C and XN40/25C, and can be divided into two distinct groups of curves for XN05/25P in the wide range of reduced time to failure, each corresponding to a different mode of fracture as mentioned above. Especially, in the range of intermediate time to failure and temperature, that is in the range of compressive fracture, the σ_f for XN70/25C and XN40/25C decrease remarkably with N_f, however the σ_f for XN05/25P decrease scarcely with N_f.

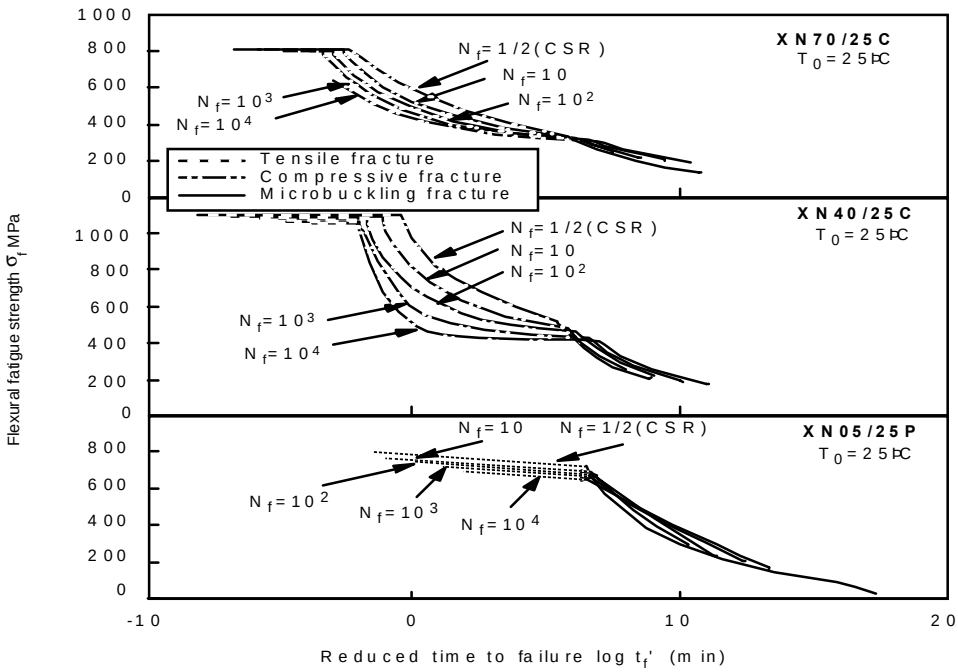

Fig.9: Master curves of fatigue strength for three types CFRP laminates.

CONCLUSION

The flexural fatigue behavior of three types of unidirectional CFRP laminates, combined with three types of pitch-based carbon fibers and single type of matrix epoxy resin, were evaluated at several levels of loading rate and temperature. The CSR (constant strain-rate) and the fatigue behavior of these CFRPs were found to be remarkably dependent on time and temperature. The time-temperature superposition principle for the CSR strength of the CFRP holds also for these fatigue strengths, although these time-temperature shift factors do not always agree with that of the viscoelastic behavior of the matrix resin. The master curves of flexural fatigue strength for three types of CFRP were obtained, and the characteristics of these fatigue strengths were clarified from these master curves.

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