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The effects of sea water and concentrated salt solutions on the fatigue of nylon 6,6 fibres

M. C. KENNEY*, J. F. MANDELL, F. J. McGARRY
Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139, USA

Cyclic fatigue and creep rupture tests have been run on high-tenacity nylon 6,6 single fibres, yarns and small ropes in air and sea water environments. Fatigue failure in each case is by a creep rupture mechanism; yarns and small ropes show the same fatigue sensitivity as do single fibres. Sea water reduces the strength by approximately 10% under most conditions. Concentrated metallic salt solutions which cause environmental stress cracking in bulk nylon do not degrade the fibres beyond the effect of plain water. Tests on oriented nylon specimens show that environmental stress crack sensitivity is greatly reduced by orientation.

1. Introduction

Many applications of nylon fibres include a complex history of environmental exposure and mechanical loading [1]. This work is concerned with nylon fibres used in marine rope, and with determining the mechanisms by which salt water environments combined with fatigue loading might cause weakening and failure. While literature on this subject is limited to a few studies of rope performance [2–4], there has been considerable interest in the areas of environmental stress cracking and moisture effects on the fatigue behaviour of bulk nylon specimens.

Environmental stress cracking in polymers refers to crack-induced failure under tensile loads in the presence of an environmental agent, which causes failure at stresses well below those that would otherwise be required [5]. The problem is generally associated with the interaction of the environmental agent with crazes in bulk polymers; specific chemical agents cause problems with specific polymers. Stress cracking of bulk nylon by salt solutions has been reported by several workers [6–9], but thus far no studies on nylon fibres have been reported. The proposed mechanisms of attack in bulk nylon involve weakening of the nylon structure by disrupting hydrogen bonding by either local plasticization and swelling (Type I salts) or dissolution of the nylon (Type II salts). Disruption of hydrogen bonds may also be caused by plain water.

Plasticization increases segmental mobility and reduces Young's modulus. Ultimate elongation generally increases in bulk specimens [10, 11], but in more highly oriented film and fibre specimens the increase may be quite small, only about 5%. The ultimate tensile strength of nylon fibres is reported to decrease about 15% in water [12]. The creep rate of bulk nylon increases with moisture, which can cause shorter failure times [10].

Few studies have combined environmental exposure and fatigue loading. One such study on bulk nylon showed a minimum fatigue crack growth rate at around 2% moisture, apparently due to embrittlement below this value and excessive softening above [13]. A study on fibres in tap water showed that strength and fatigue lifetime were both reduced by immersion; testing in this case involved biaxial rotation over a pin, which may be subject to additional complications [14].

Several studies on the behaviour of nylon ropes

Present address: Albany International Research Co., Dedham, MA 02026, USA.

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in sea water have been reported. In one [4], strength decreased by 50% and strain to failure increased by 50%. Wet ropes were also reported by Paul [2] to show greater elongation under equivalent loads, and immersion appeared to reduce heat build-up during cycling. However, no specific data were given.

In the present study, well characterized ambient fatigue data were first established and then used as baselines for comparisons with subsequent environmental effects. Detailed data for fatigue effects in air have been presented in an associated paper [15]. Emphasis in this study is on properties at the single fibre and yarn levels under creep and fatigue loading in salt solutions. Some results on small ropes and bulk materials are also reported.

2. Experimental procedures

Fatigue testing was conducted using closed-loop servohydraulic machines. Most tests were run in the load control mode, using a sine wave function. Ultimate tensile load was evaluated using a ramp test to failure at a rate equivalent to the loading portion of the fatigue cycle. Fatigue testing was conducted to various percentages of this ultimate tensile load. The specimens were cycled between the maximum tensile load and one-tenth of this value, giving a stress ratio, R, of 0.1. Other details of the test procedure can be found elsewhere [15].

A master frequency of 1 Hz was used for most testing, the master frequency referring to the frequency corresponding to cycling at 100% of ultimate tensile load. The actual test frequency at each percentage of ultimate tensile load was then adjusted upwards to maintain a constant loading rate. Thus, the frequency was increased as the maximum load was decreased. Test frequency was kept as close as possible to this level, except where machine resonances required slightly lower values. The master frequency is given on each figure.

A cardboard tabbing system was used for testing of dry yarns and single fibres. A flexible silicone sealant was used at the tab end to improve stress transfer and to protect the specimen from abrasion. Further into the tab a rigid adhesive (Eastman 910) was used. For sea water testing, a modified system was constructed of PVC tabs, which were sanded, thinned at the tip and cleaned with acetone prior to fibre bonding with Epon 828/TETA epoxy adhesive. Using this tabbing method, the majority of fibre breaks occurred in the gauge section, away from the grips, and were considered to be valid test results. For rope gripping, silicone and polyester adhesives were used to encapsulate rope ends into tapered metal fittings. The gauge length between adhesive points for single fibres and yarns was 5 inch (127 mm); the gauge length between fittings for small ropes was 4 inch (102 mm).

Control tests for comparison with various environments were run dry, in laboratory air. The ambient conditions were air conditioned without humidity control for all tests, with the exception of single-fibre creep rupture tests which were run at 65% relative humidity (RH) in a chamber. Possible effects of varying ambient humidity were investigated by conducting a series of yarn creep rupture tests at 65% RH and in ambient air, the latter tests covering both summer and winter conditions. Creep rupture lifetimes were virtually identical for the two test series over the complete load range of interest, giving lifetimes from 10 to 10^7 sec. Thus, ambient air tests give results representative of a constant 65% RH over the lifetime range discussed in this paper.

ASTM standard formulation D1141 was used for sea water composition [16]. A specially designed plastic chamber and grips were used for sea water fatigue of single fibres and yarns. During cycling the chamber is mounted on the testing machine piston and moves up and down around a freely travelling rod attached to the load cell. This configuration is necessary when using sensitive load cells that cannot support the weight of the chamber. Careful adjustment of the electronic gain is required for smooth operation, and the upper grip must remain above water to avoid control problems from hydrodynamic effects. For testing of small ropes, a large plastic tube system was used with tap water flowing through the apparatus constantly.

Creep rupture lifetimes for fibres and yarns were measured using a dead load arrangement. Environmental exposure during creep rupture was accomplished by enclosure in small heat-sealed plastics bags left open at the top for access.

In environmental tests on bulk nylon tensile bars, a small cup was formed on the (vertical) specimen surface using a bead of RTV silicone, and several drops of the environmental agent were applied. Several such cups could be formed

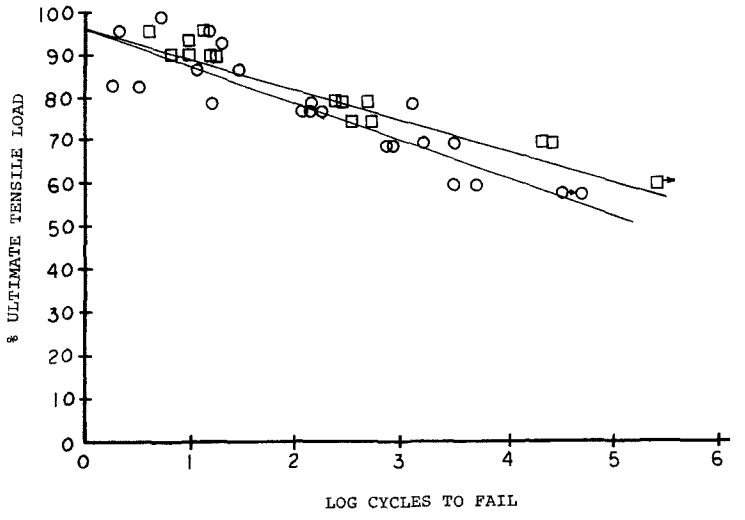

Figure 1 S–N fatigue data (1 Hz, $R = 0.1$) for single nylon fibres in air (□) and sea water (○).

on each specimen. Except for very short lifetime tests, the specimen was loaded for approximately 30 sec prior to application of the agent, as is commonly done [7, 8]. The control condition used plain water in one of the cups on the specimen. In all cases where failure was observed, the section containing the environmental agent was the source of the failure.

The primary material was DuPont 707 nylon-6,6 yarn, a standard rope yarn used commercially. The yarn consists of 210 lightly interlaced fibres producing a total of 1260 denier taken from a single lot and merge. Single 30 μm fibres for testing were carefully extracted from sections of this yarn. The small rope used was 3/16 inch (4.75 mm) diameter double braided, based on DuPont 707 nylon yarn, purchased from Sampson Ocean Systems.

Three types of bulk specimens were used to study the effect of increasing degrees of orientation. These were, in order of increasing orientation, specimens machined from large centre-gated injection-moulded plaques (surfaces removed), end-gated injection-moulded tensile bars and specimens cold-drawn from moulded tensile bars. Drawing was conducted at a slow rate (2 mm min^{-1}) on a universal testing machine at room temperature. The draw ratio was determined by the change in cross-sectional area, ranging from 2.59 to 2.83. Moisture content prior to testing was 1.4% for the machined plaques and 2.0% for the tensile bars.

3. Results and discussion

3.1. Fatigue in sea water

S–N data for short-term sea water exposure of single fibres and yarns are shown in Figs 1 and 2. These tests were conducted on specimens

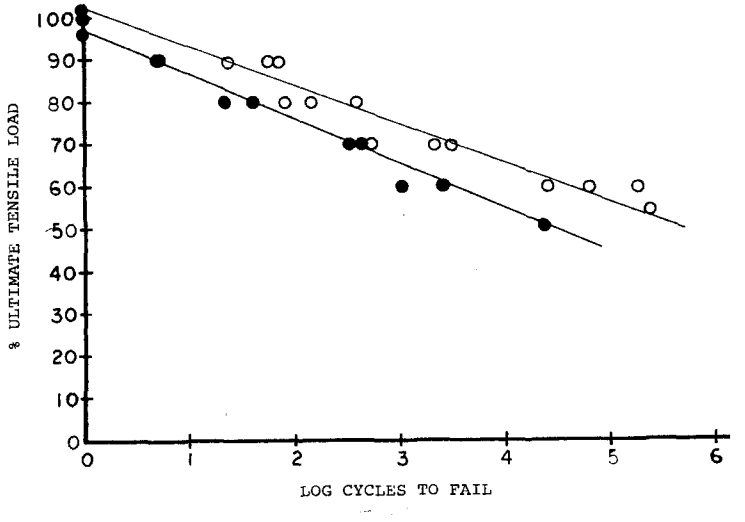

Figure 2 S–N fatigue data (1 Hz, $R = 0.1$) for nylon yarns in air (○) and sea water (●).

TABLE I Ultimate load and elongation in single-cycle ramp tests

		Ultimate* load	Ultimate* elongation (%)
Fibre	Air	68.1 g (± 2.0)	15.0 (± 0.75)
	Sea water	60.4 g (± 3.2)	14.4 (± 1.2)
Yarn	Air	11.61 kg (± 0.43)	17.0 (± 1.4)
	Sea water	12.11 kg (± 0.26)	17.8 (± 1.4)
Rope	Air	605 kg (± 21)	40.9 (± 2.0)

*Approximate failure time = 0.5 sec.

immersed in sea water for 5 min before testing. (Longer exposure times will be discussed later.) The load coordinate on each plot is normalized by the single-cycle ultimate tensile load at the conditions of that particular data set. Values of ultimate tensile load are given in Table I and can be used to determine absolute load values for the *S–N* data. Because of machine resonances with the sea water chamber, single-fibre data were run in the range 0.6 to 1.0 Hz and yarn data in the range 1.1 to 1.2 Hz.

The effects of sea water immersion during fatigue are relatively mild. Lifetimes at the lower stress levels in sea water are displaced from those in air by less than one decade shorter lifetime or 10% lower (normalized) load. Although a ten-fold decrease in lifetime is significant, it represents only a small shift in the failure load. This does not appear sufficient to be a dominant factor in those service failures that occur at relatively low loads. The initial ultimate tensile load for the fibres, as given in Table I, also drops by about 10%, as has been observed in the literature [12]. Initial ultimate tensile load for the yarn increases very slightly on short-term sea water exposure. This may be explained by a lubricating effect of the water, which allows fibres within the yarn to align in a more nearly parallel, more efficient, structure.

S–N data for short-term tap water exposure of a limited number of specimens of small rope are shown in Fig. 3. The data for wet and dry ropes are similar to those of the other structures; the *S–N* data in sea water are reduced to about 10% lower load or one decade shorter lifetime. In this case, however, both the data for wet and dry ropes are normalized to the initial strength of the dry rope, and run at 0.5 Hz. Grip failures became a problem at lower loads, and are shown in Fig. 3 by points with arrows, representing the lowest possible lifetime for gauge section failure.

Sea water results for single fibres, yarns and small ropes are compared in Fig. 4. The results are plotted as a function of log time rather than cycles to account for differences in the frequencies used. Single-fibre and yarn data are approximately linear, with similar slopes, and nearly superimpose on a normalized scale. This can also be seen from comparison of the regression equations, where 95% confidence limits are given in parentheses:

$$\text{single fibre } P/P_{ult} = 1.03(\pm 0.44) - 0.103(\pm 0.003)\log N$$

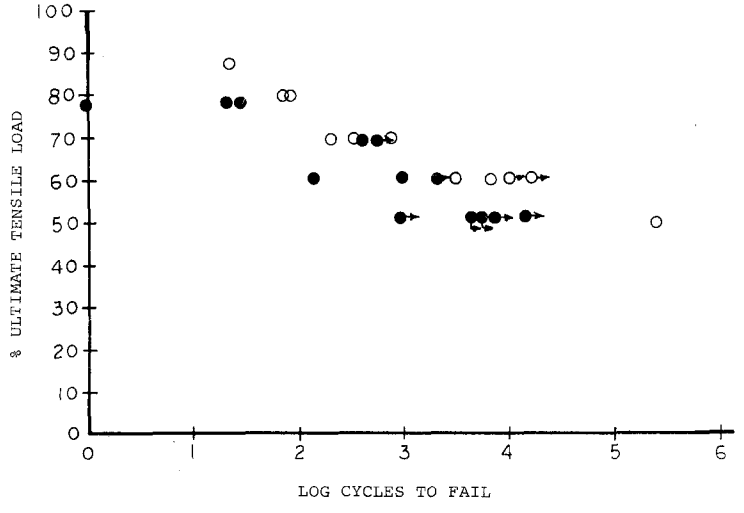

Figure 3 S–N fatigue data (0.5 Hz, $R = 0.1$) for 3/16 inch nylon double-braided rope in air (○) and tap water (●) (normalized by dry ultimate load).

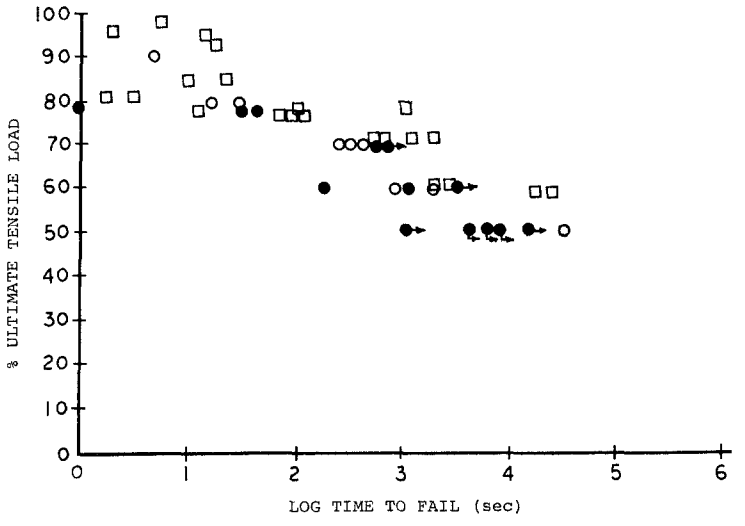

Figure 4 Comparison of fatigue data for single fibres, yarns and ropes in water: □, fibre, 1 Hz; ○, yarn, 1 Hz; ●, rope, 0.5 Hz.

$$\text{yarn } P/P_{ult} = 0.972(\pm 0.03) - 0.111(\pm 0.002)\log N$$

where P is the load and N is cycles to failure. Small-rope data are generally similar to those of single fibres and yarns, but the occurrence of many grip failures limits the significance of comparisons. The similarity of normalized data among the various structures indicates a common factor in their fatigue degradation, as has also been shown [15] for dry structures.

The associated paper [15] on fatigue in air considered the origin of the cyclic fatigue behaviour. It was shown that a creep rupture-based model could accurately predict the cyclic fatigue lifetime over a range of conditions. Compared with a constant maximum load in a creep test, cycling to lower loads while maintaining the same maximum load simply extends the total time to failure by reducing the time at high loads. Failure was shown to occur at a particular cumulative strain for each level of structure regardless of the type of loading. Over a broad range of frequency, failure occurred at a particular total test time, independent of the number of cycles.

Fig. 5 and 6 give creep rupture data for single fibres and yarns in sea water, air and other environments, which will be discussed later. The load data in Fig. 6 are normalized by the ultimate tensile strength in that environment from a ramp test having a failure time of approximately 0.5 sec (Table I), while the data in Fig. 5 are not normalized (the ramp values are indicated on the load coordinate). Creep rupture lifetimes for the single

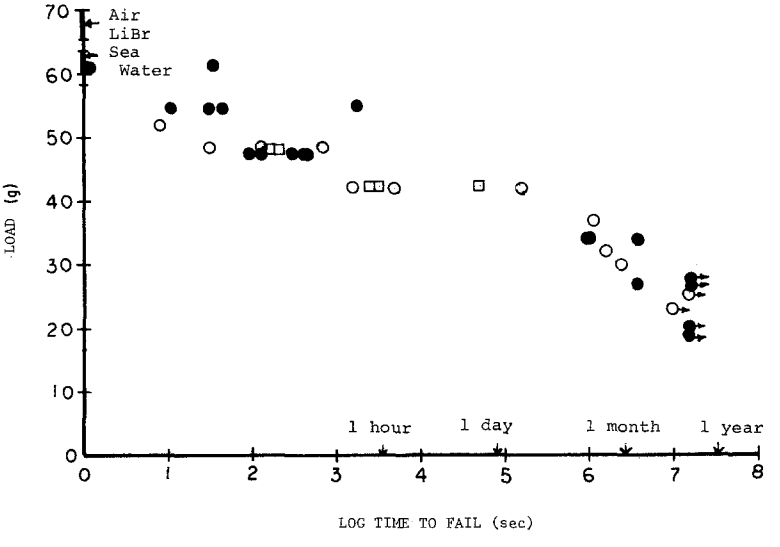

Figure 5 Comparison of creep rupture data for single fibres in several environments: ●, air; ○, sea water; □, LiBr (40 wt %).

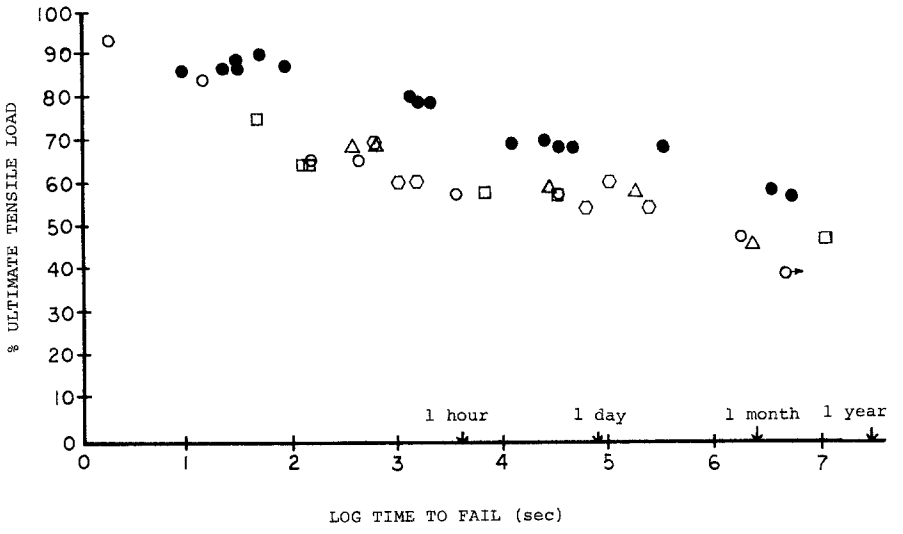

Figure 6 Comparison of creep rupture data for yarns in several environments: •, air; ○ sea water; △, 20% LiCl; ○, 40% LiBr; □, deionized water.

fibres are generally similar in air and sea water, at the same load. For yarns, the creep rupture curve in sea water falls at 10 to 15% lower load than that in air, but has a similar trend. The yarns have about the same strength in sea water and air (Table I) despite the lower individual fibre strength in sea water; this is apparently the result of a greater bundle efficiency in water, possibly due to a lubricating effect. Thus, the effect of sea water on the yarn creep rupture curve may be due not to a different response to creep, but to the initial ramp strength used to normalize the data; the dry strength and creep rupture are complicated by broken fibres which only partially unload [15].

The creep rupture data in Fig. 5 and 6 have been used to predict the cyclic fatigue response through the model described earlier [15]. Fig. 7 and 8 show that the cyclic data are in general agreement with the lifetime ranges predicted from creep rupture. In fact, the wet yarn data are in better agreement than those tested under dry conditions [15]. The water environment apparently lubricates the yarn and precludes complications from broken fibre reloading observed in the creep rupture behaviour of dry yarns. Fig. 7 and 8 clearly demonstrate that the cyclic fatigue of nylon 6,6 single fibres and yarns in sea water as in air is due to a creep rupture mechanism. Table II

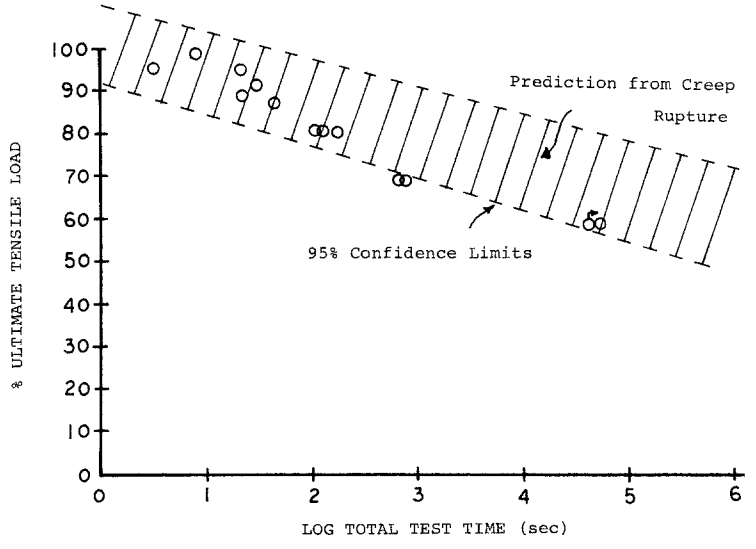

Figure 7 Predicted and experimental fatigue data for single fibres in sea water.

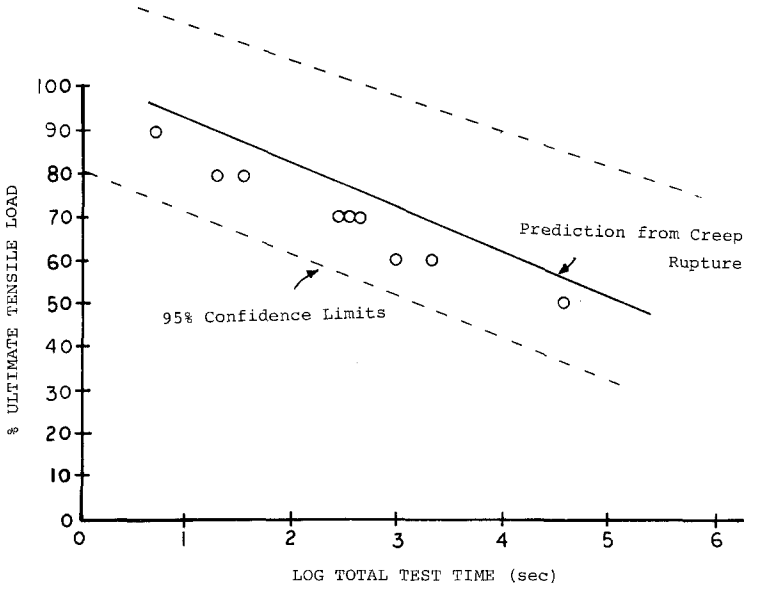

Figure 8 Predicted and experimental fatigue data for yarns in sea water.

shows that the cumulative strain to failure is the same whether the tests are creep rupture, cyclic fatigue or simple ramp tension, also consistent with the creep rupture model.

3.2. Effects of preconditioning under load

The fatigue data in Fig. 1 to 3 are for tests with only a 5 min preconditioning time in sea water. Selected tests have also been run with longer preconditioning while under a dead load. For yarn specimens immersed in sea water for one month at dead loads of 14% and 3% of the wet yarn strength, and for control specimens immersed unloaded, a very slight decrease in ramp tensile strength with increasing applied load during conditioning was observed. The strength decreased from 11.99 kg in the unloaded case to 11.47 kg for the 14% loading (Table III). However, the overall effect is minimal, and traditional environmental stress cracking does not appear to occur on this timescale. Specimens loaded at 3% in air for one month showed a slight increase in tensile stength. This effect is attributed to an initial structural alignment in the yarn, as similar increases are observed in specimens loaded for only 5 min periods. Several specimens were also soaked in sea water and allowed to dry; this was repeated three times in succession with no adverse effects on dry strength.

Fatigue data obtained after the four types of one month conditioning are plotted in Fig. 9. All sea water behaviour, including the original 5 min immersion data, fall on approximately the same line. Clearly, there is no significant effect of this conditioning on fatigue or strength. Data from specimens which were preloaded and tested dry also follow the original dry S–N curve.

TABLE II Comparison of elongation at break for single-cycle ramp, fatigue and creep tests

		Cumulative strain to failure (%)		
		Ramp	Fatigue*	Creep*
Fibre	Air	15.0 (± 0.75)	14.6	14.4
	Sea water	14.4 (± 1.2)	14.5	14.9
Yarn	Air	17.0 (± 1.4)	18.9	18.9
	Sea water	17.8 (± 1.4)	17.6	–
Rope	Air	40.9 (± 2.0)	41.9	41.4

*Single specimen at 70% of ultimate load, typical data.

TABLE III Residual ultimate strength and elongation of DuPont 707 nylon yarn after one month conditioning

Conditioning load, Environment	Ultimate load (kg)	Elongation (%)
Unloaded, Sea water	11.99 (± 0.20)	21.8 (± 0.5)
1.7 kg, Sea water	11.47 (± 0.29)	17.4 (± 0.4)
0.4 kg, Sea water	11.65 (± 0.28)	18.3 (± 1.0)
0.4 kg, Air	12.40 (± 0.49)	16.2 (± 0.9)

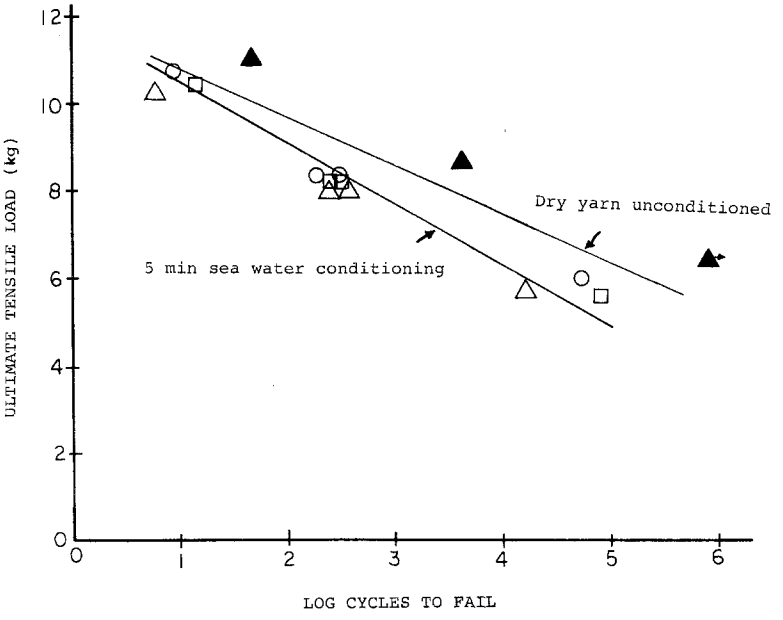

Figure 9 Fatigue data for yarns after one month conditioning under several loads and environments: ○, unloaded, sea water; △, 1.7 kg, sea water; □, 0.4 kg, sea water; ▲ 0.4 kg, air.

In view of the minimal effects observed after one month of sea water conditioning, a considerably longer period of immersion under load was studied. After immersion for ten months under 19% of ultimate load, failure at or quite near the grips during subsequent testing was a problem; however, even these tests gave an average strength of 80% of the initial strength.

3.3. Effects of more severe stress cracking agents

The standard sea water solution is very dilute and the components relatively inactive when compared to the aqueous solutions of certain salts which have been reported to stress crack bulk nylon [7, 8]. To investigate environmental sensitivity further, series of tests were conducted in more potent stress cracking agents: solutions of LiBr (40 wt%) and LiCl (20 wt%). Saturated solutions of these salts have been reported to crack bulk nylon within several minutes to 48 h [7, 8]. In the current tests, specimens were immersed in the solutions at several load levels, and creep rupture lifetimes were recorded. Yarn results, plotted in Fig. 6, show that LiCl, LiBr and also plain deionized water have exactly the same effect as standard sea water, showing approximately 10% lower loads or about one or two decades shorter lifetimes compared with dry specimens. The common behaviour observed in all aqueous solutions clearly shows that the dominant factor is the aqueous environment, with no measurable effect of the other constituents. It appears that the process is controlled by simple plasticization, as has been observed for nylon in an aqueous environment [10]. Tests on single fibres (Fig. 7) gave similar results.

3.4. Effects of moisture on stress–strain behaviour

The effects of water on nylon under the conditions of this study appear to be a simple plasticizing action. Fig. 10 gives simple constant displacement rate stress–strain curves of yarns in ambient air and sea water after a 5 min conditioning period (the rate is equivalent to a 1 Hz test). The effect shown is completely reversible: if the yarn is immersed and then redried, the original dry stress–strain curve is again obtained. The initial modulus of the yarn in water is only 38% of the value in ambient air, while the remainder of the stress–strain curve and the failure conditions are much less affected. Tests on single fibres and yarns after as much as four months immersion in sea water show no further reduction in modulus beyond the effect at 5 min.

3.5. Effects of orientation on environmental stress cracking resistance

Textile fibres appear to be less susceptible to stress cracking agents than are bulk nylon specimens. The effect of orientation was studied directly using bulk nylon 6,6 specimens containing varying

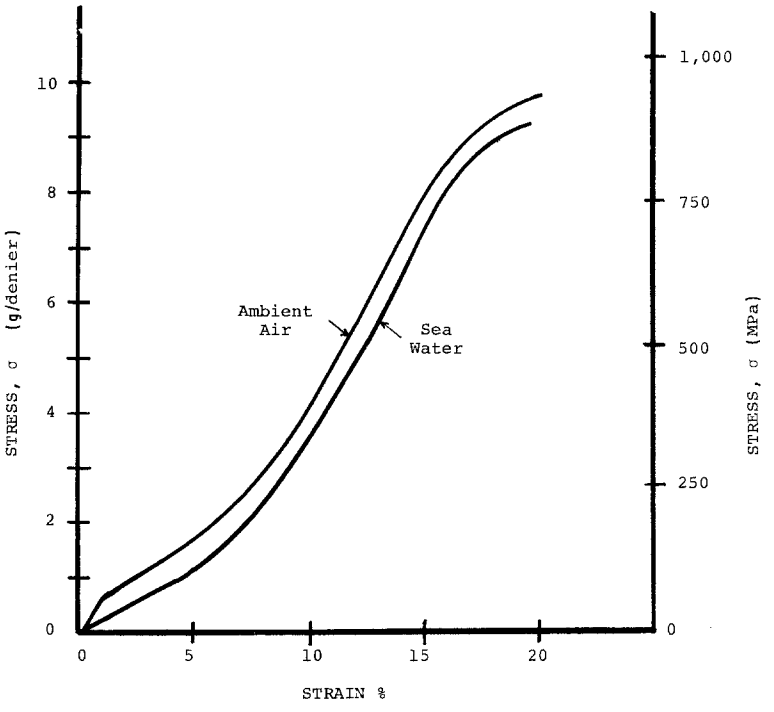

Figure 10 Typical stress–strain curves for yarns in ambient air and sea water after 5 min conditioning.

amounts of orientation produced by methods described earlier. Wide-angle X-ray diffraction patterns of the three bulk specimen types are shown in Fig. 11. The two moulded, undrawn specimens show little orientation; they differ primarily in surface orientation, which is not distinguishable in diffraction patterns averaged through the thickness. Cold-drawn tensile bars show a notably increased orientation, indicated qualitatively by the bright diffraction spots as compared with uniform rings for the unoriented materials. Test results for bulk specimens are summarized in Fig. 12. Ultimate tensile strengths were determined for each specimen type, and stress cracking tests were conducted at various percentates of the ultimate. Ultimate tensile strength, based on the initial cross-sectional area after drawing, ranged from 10 900 to 29 000 lb in^{-2} (75.1 to 205 MPa) for the three increasing levels of orientation. For the machined plaques, failures with the cracking agent present were brittle at all load levels, apparently from crazing induced by the LiBr solution. All failure times are actually quite long (greater than 2500 sec), showing that even in this aggressive environment the bulk specimens do not degrade rapidly. The failure times were longer than in reported studies [7], possibly the result of differences in specimen type and test conditions.

The injection-moulded bars show consistently longer failure times than do the machined plaques, except at high stresses where ductile failure becomes the dominant mode. For the drawn specimens, failure times are still longer, both at the same absolute stress level and at an equivalent percentage of ultimate tensile strength. Drawn specimens crazed or cracked extensively, but did not separate.

It is apparent that increasing amounts of orientation are effective in suppressing environmental stress cracking. Therefore, high-tenacity fibres of even greater orientation and ultimate tensile strength would be expected to show still less sensitivity to stress cracking agents. Long-term tests on fibres, still in progress, show no evidence of environmental stress cracking after two years at 40% of ultimate load.

The suppression of environmental stress cracking can be understood in terms of the fibre structure and mechanism of cracking. Conventionally, environmental stress cracking occurs by formation of a craze perpendicular to the stress direction, which allows subsequent penetration of the environment by capillary action [17–19]. Orientation of polymer chains parallel to the fibre axis, and thus the stress direction, makes formation of perpendicular crazes increasingly difficult. Crazes, if formed at all, must occur parallel to the stress

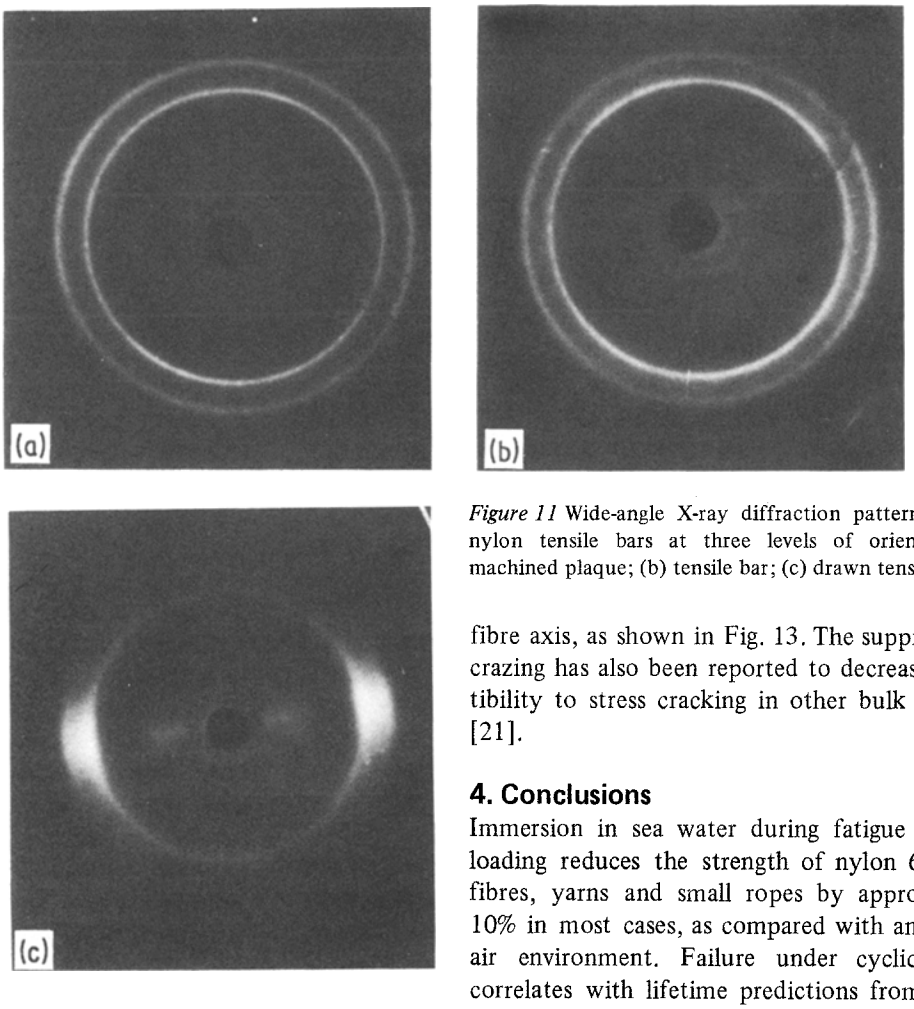

Figure 11 Wide-angle X-ray diffraction patterns of bulk nylon tensile bars at three levels of orientation: (a) machined plaque; (b) tensile bar; (c) drawn tensile bar.

direction in a much more difficult process. Some examples of crazes of this type have been observed in the literature [20]. In the tests on fibres reported here, only one case of crazing has been found; this apparent craze runs parallel to the fibre axis, as shown in Fig. 13. The suppression of crazing has also been reported to decrease susceptibility to stress cracking in other bulk polymers [21].

4. Conclusions

Immersion in sea water during fatigue or creep loading reduces the strength of nylon 6,6 single fibres, yarns and small ropes by approximately 10% in most cases, as compared with an ambient air environment. Failure under cyclic loading correlates with lifetime predictions from a creep rupture-based model. Concentrated solutions of LiCl and LiBr, which cause environmental stress cracking in bulk nylon, have no effect on fibres beyond the effects of pure water. The increased resistance of fibres results from orientation

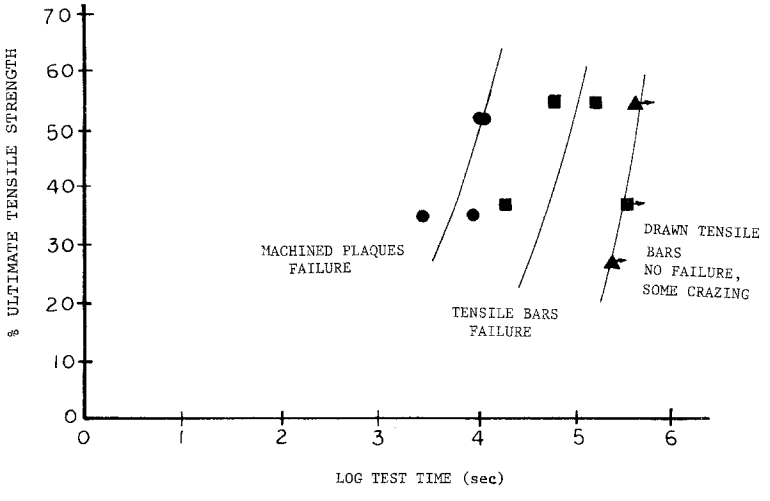

Figure 12 Environmental stress cracking in LiBr solution for bulk nylon at three levels of orientation.

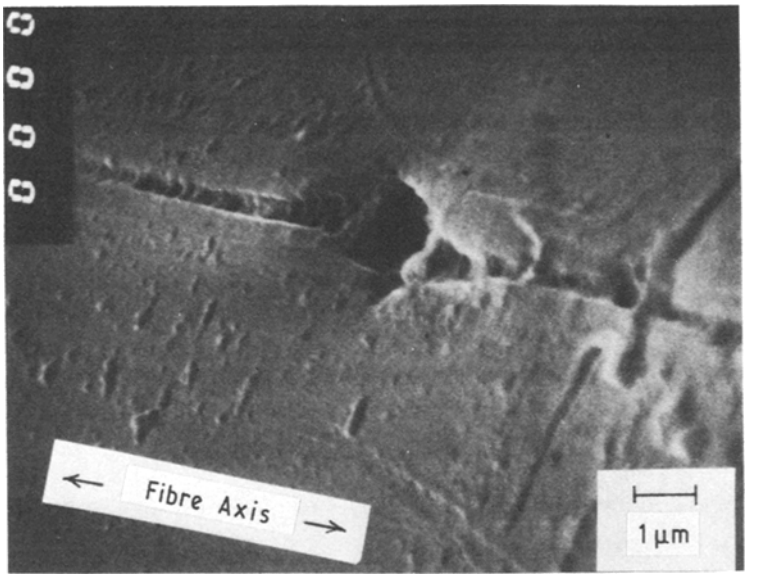

Figure 13 Craze structure parallel to axis of fibre exposed to LiCl solution (20 wt %).

introduced during drawing from bulk to fibre form.

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