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DURABILITY OF ARAMID ROPES IN A MARINE ENVIRONMENT

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ABSTRACT

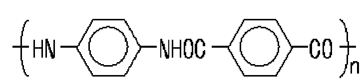
This paper presents first results from a study of the long term marine durability of aramid fibers. The program was started based on the experience of IFREMER using aramid fiber ropes for instrumentation and deep sea handling lines, which is described here. Instrumentation lines showed no degradation after recovery, but results from testing of handling ropes after service indicated significant strength reduction. This led to the development of specific test facilities to study bend-over-sheave performance. The overall aim is to improve understanding of the roles of both the fiber and the rope construction in a marine environment, in order to improve long term strength retention of aramid marine ropes.

Keywords: aramid, braid, aging, bending fatigue

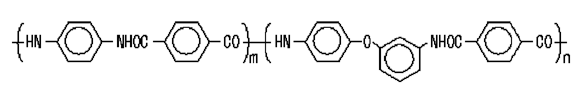
INTRODUCTION

High performance ropes and wires based on aramid fibers have been used for over thirty years for marine applications such as handling lines, buoy and scientific instrument mooring lines, pendant lines on drilling platforms,…[1]. Their high tensile strength, low creep and elongation, light weight, and high chemical resistance make them a serious competitor to steel wires, but their poor compression resistance requires special care during handling.

Two aramid fibers which can be used for marine applications are the *Twaron* and *Technora* grades, both produced by Teijin, which differ in their chemical formula [2,3], their structure and morphology [4-11]. The *Twaron* fibers are based on poly(*p*-phenylene terephthalamide) (Figure 1). The *Technora* fibers are based on co-poly-(paraphenylene/3,4'-oxydiphenylene terephthalamide) (Figure 1).



poly(p-phenylene terephthalamide) (Twaron®)



co-poly-(paraphenylene/3,4'-oxydiphenylene terephthalamide) (Technora®)

Figure 1. Aramid molecular structures

Several previous studies have presented data on the durability of aramid fibers and a brief overview of these studies will be presented first. Then the first results from the current project on aging of fibers will be given, followed by some results from in-service experience with aramid ropes. The paper will conclude with some preliminary results from tests on a fiber with improved marine finish, specifically developed for long term marine applications.

PUBLISHED WORK ON ARAMID FIBER DURABILITY

Several tests have been performed to evaluate the tensile creep durability of aramid fibers. For example, Lafitte [12] studied the influence of the applied creep load on the life time of *Kevlar 29* fibers. At 70% of the failure load, about 75% of the fibers broke after 60 hours [12] while no ruptures were observed at 56% of the failure load. Riewald [1] reported that the strength of *Kevlar 29* and *Kevlar 49* did not decrease after 90 days loaded at 40% of the failure strength. Morgan *et al*. [14] suggested that the strength and stress-induced aging of *Kevlar 49* are related to microvoid growth and the generation of new microvoids under applied load. Other authors [15,16] describe the creep strain from a microstructural point of view. Ericksen [15] separated the crystallite rotation into two mechanisms : the elastic strain of the crystal lattice and the plastic strain by shifting of the crystallites. Northolt and colleagues also studied these effects in detail [16] and extended a continuous chain model to include viscoelastic effects. Chailleux applied a non-linear viscoelastic model to successfully predict strains in *Twaron 1000* yarns subjected to different load sequences [17].

Tension-tension fatigue tests have been carried out to evaluate the durability of fibers subjected to cyclic loadings. Lafitte and Bunsell [13] gave some interesting qualitative results, reporting that the life time of *Kevlar 29* single fibers subjected to tension-tension fatigue decreases when either the maximum load or the amplitude is increased. Depending on the maximum applied load and the amplitude of the imposed strain, these authors distinguished two different failure modes under cyclic loadings, either creep or fatigue failures.

From a chemical point of view, hydrolytic degradation has been considered. The hydrolysis mechanism of poly(*p*-phenylene terephthalamide) (PPTA) fibers presented by Morgan *et al.* [14] involves homolytic scission of the amide N-H linkage, yielding acid and amine functions. The authors established a relation which shows that the fiber strength degradation increases with time, temperature and applied load [14]. The average strength loss is 0.6% per year for unloaded fibers in a 100% relative humidity environment at 23°C, and 3.2% per year for fibers loaded to 75% of their failure strength under the same conditions [14]. Riewald [1] reported that yarns loaded at 35% of their failure strength in sea water and under a pressure of 55 MPa, displayed a strength loss of 2% for *Kevlar 29* and 0% for *Kevlar 49* after 90 days exposure. The strength of these yarns decreased by 1.5% after one year exposure in sea water [1]. Springer *et al*. [18] showed that the addition of 10 % NaCl to pure water reduced the degradation of *Kevlar 49* fibers due to a “shielding” effect, whereas it did not change for *Twaron T1055* fibers. The authors also followed the effects of basic and acid solution exposure (respectively 10% NaOH and 40% H_2SO_4) on mechanical properties. Both modulus and tensile strength are affected under wet aging, but the property drops are larger in acid and basic solutions than in a neutral environment.

MATERIALS CONSIDERED

Two materials have been chosen for the present aging study: *Twaron 1000* fibers in the form of 1680 dtex yarns, and *Technora T200W* fibers in the form of 1670 dtex yarns. They are both produced by Teijin Aramid. The former is glossy with a yellow colour, the latter is beige and matt.

Two types of aramid rope will be considered here. The first is a small 3 ton break load parallel fiber aramid line used for holding instrumentation equipment at different sea depths. The second is a braided aramid rope used for deep sea handling, of break load 20 or 50 Tons. More details will be given in the following sections.

FIBER AGING

Aging environments

Three different aging environments are considered in this study. The first aims at studying the durability of aramid fibers in a marine environment. Four natural sea water tanks are heated to 20, 40, 60 and 80°C. The pH value was measured to be between 8.0 and 8.6 at 14°C, and the salinity between 32.8 and 33.4 g/L. A pumping system renews the sea water continuously. In the same way, four deionised water tanks are heated to 20, 40, 60 and 80°C in order to evaluate the effect of pure water only. Their pH value was measured to be between 7.0 and 7.2 in the 14.0-14.5°C range. Figure 2 shows the eight tanks.

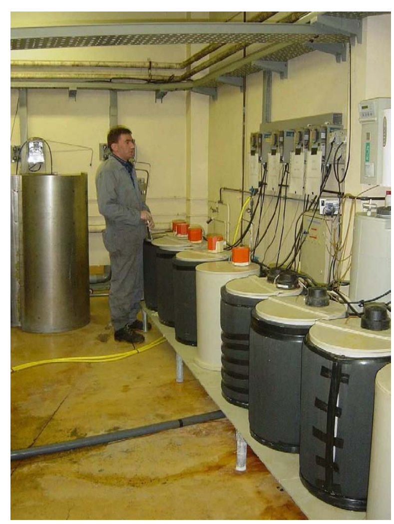


Figure 2: Temperature controlled circulating sea water and pure water tanks

A second series of samples has been aged at pH9 and pH11 in sodium carbonate salt solution, at temperatures from 20 to 80°C. These are primarily intended to simulate civil engineering applications, but may also be of interest for coastal infrastructure.

A third series is aged under tensile load, both in air and sea water. Loads are introduced by dead weights via a pulley system. Initial loads were 40 and 60% of the fiber failure strength. Again a pump system circulates the water. The time to failure is noted. Figure 3 shows the test frame.

Figure 3: Sea water bed test with static loads

Analysis, characterization and mechanical testing techniques

Several techniques have been used to follow thermal, physico-chemical, chemical and mechanical properties. Morphology changes are also characterized. First results and some examples for *Twaron 1000* will be shown below, but it should be emphasized that this part of the work is ongoing and will continue for at least another year.

FTIR spectrometry

In order to follow the evolution of chemical functions at different aging times, the intensities of characteristic peaks are followed by Fourier Transformed InfraRed spectroscopy using a Diamond ATR (Attenuated Total Reflectance) apparatus. The spectra are obtained from a *Nicolet Impact 410* spectrometer. The data are analyzed with *OMNIC 3.1* software. Fifteen fibers are taken for each trial, each trial is repeated three times.

A peak related to the fiber finish and located at 1109 cm^{-1} has been identified for *Twaron 1000* fibers. Another stable characteristic peak has been chosen to calculate the relative intensities: the peak located at ~821 cm^{-1} related to the C-H deformation of aromatic rings, appears to be appropriate. An example of results is shown in Figure 4.

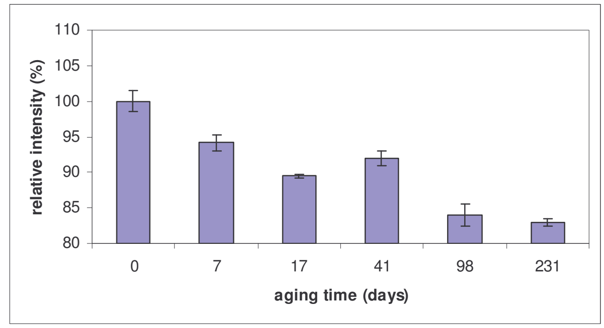


Figure 4: Relative intensity change of the peak related to the finish for *Twaron 1000* fibers, pure water, 80°C.

A significant decrease of the ratio of intensities is observed after 3 months in pure water at 80°C. The ATR can thus be used to reveal changes in surface finish under these aging conditions.

The relative intensities of the amide function characteristic peaks have also been followed for *Twaron 1000* fibers. The first is located at ~1634 cm^{-1} related to the C=O, the second at ~1538 cm^{-1} related to the combined motion of N-H bend and C-N, and the last at 1307 cm^{-1} related to the C-N, N-H and C-C combined vibrations [19]. After 231 days in pure water at 80°C, no clear trend can be drawn. The hydrolytic degradation scheme for PPTA given by Morgan *et al.* [14] would indicate that amide functions decrease, and acid and amine functions increase. A possible explanation for the observed stability of the amide characteristic peaks is that the macromolecules degraded at the surface of the fibers leave in the solution. Further aged samples will be analyzed to explore this.

Density measurements

Density experiments are performed to examine whether pores develop within the fibers. Initially 0.1 g of material is weighed at 20°C in ambient air. Then the fibers are weighed after immersion in dodecane, which appears to be inert with the *Twaron* and *Technora* fibers. The densities of air and dodecane are both known, so the density of the fiber can be calculated. Density is then measured periodically throughout the aging period.

For *Twaron 1000* fibers aged in pure water, the density first increases with aging time from 1.38 to 1.44 after 41 days at 20°C, and from 1.38 to 1.42 after 7 days at 80°C. This increase is interpreted as mainly due to departure of finish, the density of which is lower than that of the aramid, resulting in a global increase of the density. The finish loss is thus faster at 80°C than at 20°C. Next the density starts decreasing down to 1.41 after 231 days at 20°C and 80°C, indicating that pore generation may be occurring. However, the finish loss is continuous under these conditions, as indicated by the previous FTIR results, so quantitative conclusions require further analysis. An example of the profile of the density change curves is shown in Figure 5.

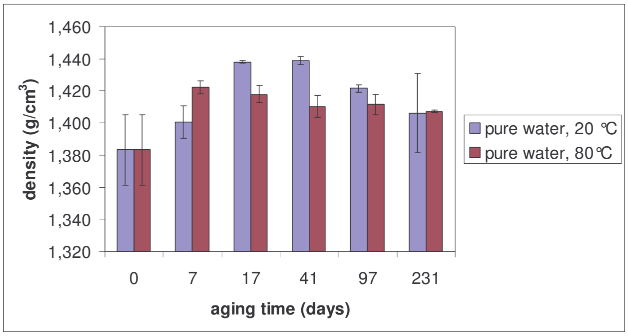


Figure 5: Density versus aging time of *Twaron 1000* fibers immersed in pure water at 20 and 80°C

Differential Scanning Calorimetry

Analysis was performed on DSC Q100 equipment (*TA Instruments*). The TA Universal analysis software is used to process the data. The experiments were carried out under a nitrogen flow of 50 ml/min. 10 mg of material are analyzed. The heating sequence is the following: the sample is heated from 0 to 400°C (below the degradation temperature) at 30°C/min. Then it is stabilized at 400°C for 15 min in order to eliminate the thermal history of the material. Next it is cooled down to 0°C at 30°C/min. Finally, the sample is heated up to 400°C again at 30°C/min.

Particular attention was paid to the glass transition temperature, previously studied by Penn and Larsen [19], and Lafitte [12] for PPTA fibers. Lafitte has shown that structural changes arise at this temperature. The *Twaron 1000* fibers thermograms display a small transition around 339°C (see Figure 6). A very small increase of the glass transition temperature (around 1°C) is noted after 231 days in sea water and pure water at 80°C. This could be attributed to a further re-crystallization of the macromolecular chains ends but more work is needed to confirm this.

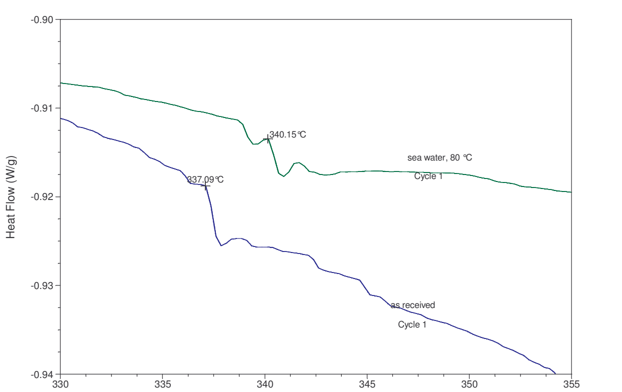


Figure 6. Glass transitions of *Twaron1000* fibers: lower, as received, upper, after 231 days in sea water at 80°C.

Thermo-Gravimetric Analysis

Thermo-gravimetry continuously measures the mass of a sample subjected to a steady increase of temperature. It enables changes in degradation temperatures, the amount of remaining finish and the absorbed water rate to be measured at different aging times. The apparatus used is a *Netzsch STA 409* TGA. The temperature of the furnace was programmed to rise at constant heating rate of 10°C/min up to 1150°C. The tests were performed under a synthetic air flow of 80 ml/min. Around 25 mg of fiber are weighed for each test. An example of a thermogram and its interpretation is shown in Figure 7.

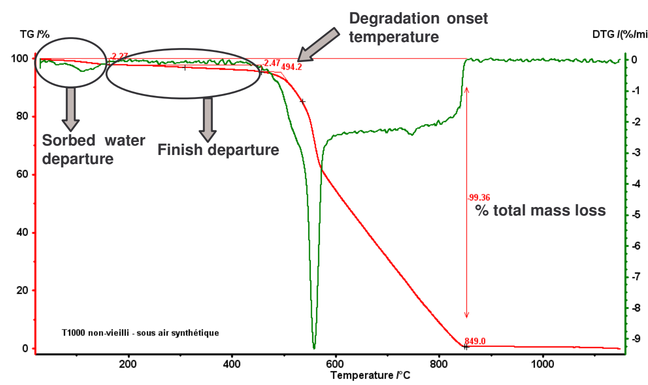


Figure 7: Profile and interpretations of thermogram obtained from as-received *Twaron 1000* fibers

The first results for *Twaron 1000* fibers show a decrease of the degradation onset temperature from 496 to 486°C after 97 days aging in sea water at 80°C, and from 496 to 490°C after 97 days aging in pure water at 80°C. These lower degradation temperatures may be the result of macromolecular chain scission, leading to a decrease in average molecular masses, and thus to smaller macromolecular segments which are degraded at lower temperature. This interpretation will be checked later by viscosity measurements. Further work is required to follow the amount of absorbed water and the finish content versus the aging time.

Viscosimetry

Viscosity measurements can indicate chain scissions. The Mark-Houwink relation given by Arpin and Strazielle in sulphuric acid concentrated at 96%, $[\eta] = 8\times10^{-3}\ M^{1.09}$ [20], enables the average molecular mass to be followed with aging time. An Ubbelhode DIN capillary viscosimeter (*Schott Instruments*) was used. To calculate the inherent and the reduced viscosity, four concentrations have been chosen: 5.10^{-4} g/ml, 10^{-3} g/ml, $1.5.10^{-3}$ g/ml and 2.10^{-3} g/ml. Higher concentrations were found to give poorer repeatability of the measurements. The material is dissolved for 2 hours at 60°C under magnetic stirring.

The first results show an important decrease of the average molecular mass M_w of *Twaron1000* fibers after 231 days in a sodium carbonate salt solution of pH 11 at 80°C. Indeed, M_w goes from 31940 g/mol to 17310 g/mol after aging. This reveals that hydrolysis and chain scissions do occur under basic exposure conditions.

These measurements will also be carried out on the fibers aged in pure and sea water, to study the influence of the pH on the hydrolytic degradation.

Scanning Electron Microscopy

Scanning electron microscopy was performed to detect surface damage induced by aging. Fibers were analyzed in the secondary electron mode, at a tension of 12 kV.

Some degradation has been observed on the surface of certain (but not all) *T1000* fibers aged in sea water at 80°C for 231 days, Figure 8. Longitudinal grooves are observed on some fiber segments and some fibrils are visible. This fibril removal, may be caused by weakening of the tie fibrils during sea water exposure but mechanical damage in the water baths due to water circulation and handling may also occur.

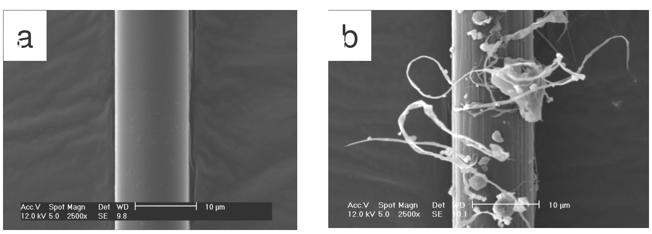


Figure 8: SEM photos of *Twaron 1000* fibers (a) as-received, (b) after 231 days in sea water at 80°C

Tensile Tests

An extensive programme of tensile testing has started in order to quantify the effects of the different environments on mechanical properties. This includes single filament testing, 30 samples for each condition, and yarn testing. Yarn samples for aging were wrapped around cylinders and placed in water. Figure 9 shows an example of yarn test results for one condition. Eight aged and eight new yarns were tested. The gauge length (length measured between the clamps) was 300 mm, loading rate was 10 mm/min.

There is a small drop in strength for the aged yarns though this may be partly due to damage introduced during handling when samples were immersed and removed from the water bath.

Dynamic mechanical analysis (DMA) on *TAI 2980* equipment has also been used to follow the changes in tensile storage and loss modulus with temperature after different aging periods.

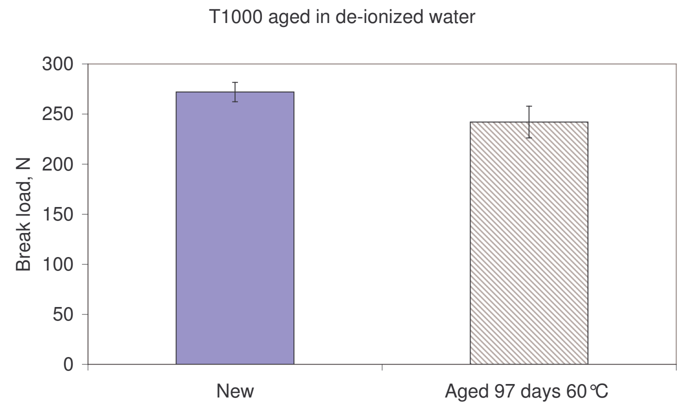


Figure 9. Tensile tests on *Twaron 1000*, 8 samples of new and aged yarns.

INSTRUMENTATION LINE EXPERIENCE

The first applications of aramid ropes at IFREMER were for instrumentation lines over 15 years ago by the Laboratory for Physical Oceanography. These are small diameter lines of 3 or 5 ton break load, onto which are attached various instruments to make measurements of parameters such as temperature and current.

The construction is parallel *Kevlar 29* aramid fiber with a protective outer coating, typically polyethylene. Figure 10 shows the end of a line with the outer cover removed over 35 mm to show the parallel fiber core.

Figure 10. Instrumentation line.

Lengths employed are typically 3 to 4 km and these lines may remain at sea for one or two years before being recovered. At IFREMER the lines are single-use as the instrumentation equipment cost is very high, but in some less critical applications the lines can be re-used several times. The loads during operation are quite low, the main loading is during deployment and recovery.

The experience with these lines has been very good. No problems have been noted with the aramid fibers, initial concerns with metallic conical end fittings were resolved by careful attention to contact surfaces.

In order to examine whether re-use of these lines resulted in a loss of strength some 700 mm lengths of re-used line were recovered, terminated with metal cones and tested, Figure 11. The used samples were either terminated directly or rinsed in tap water and dried before terminating, to remove salt residues. The plot of results from new and used specimens tested to failure at the same time indicated only one low result, which was attributed to a poor termination and not to a material degradation. Inspection of the aramid cores showed no evidence of degradation.

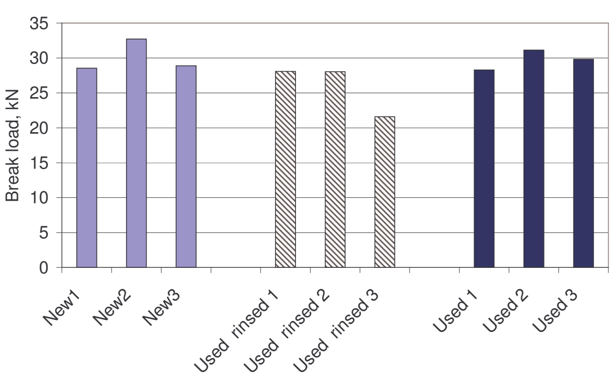


Figure 11. Tensile test on aramid instrumentation line samples new and after use. Upper, test set-up showing cone termination, lower, test results.

DEEP SEA HANDLING ROPES

IFREMER has been using aramid fiber ropes for deep sea handling for many years. A specific braided *Technora T220* aramid rope construction, Figure 12, has been developed for this application.

Figure 12. Handling rope construction showing from left to right elastomer cover, polyester braid and aramid core.

The application involves lowering equipment weighing up to 5 Tons to the sea floor, at depths down to 6000 meters, performing operations such as coring or dredging, then lifting samples back up to the oceanographic vessel. In the case of coring the sample must be extracted from the seabed, requiring additional load capacity. End terminations may be cones or splices, depending on the equipment to be handled.

Test development for handling ropes

In order to test these deep sea handling ropes it has been necessary to develop special test machines. First, a 100 ton test frame was designed and built, in order to be able to characterize the stiffness, strength, creep and cyclic behavior of aramid ropes before and after service. The test frame is 9 meters long, Figure 13 and the loading piston is controlled by a servo-hydraulic system piloted by a *Zwick 7500* controller. Specific software allows loading sequences to be programmed through a PC interface, so complex load cycles can be defined and run in a reproducible manner. Two digital cameras and in-house image analysis software are used to measure strains accurately.

Figure 13. 20T break load *Twaron 1000* aramid rope on 100 ton capacity test frame during strain calibration trials.

In order to study the response of ropes to repeated bending over sheaves (BOS) a second machine was designed and built, Figure 14. This is a test frame with three hydraulic pistons capable of applying a constant tension up to 50 Tons and then imposing a back-and-forth motion over the sheave.

Figure 14. Bend-over-sheaves test machine specifically designed to test ropes on full size oceanographic sheaves up to 2 meter diameter.

Degradation In service of handling ropes

In order to quantify the changes in rope properties after service 8 meter long samples were cut from the 20 ton break load handling lines, spliced and tested to failure in tension. Figure 15 shows the test results compared to four new rope sample strengths.

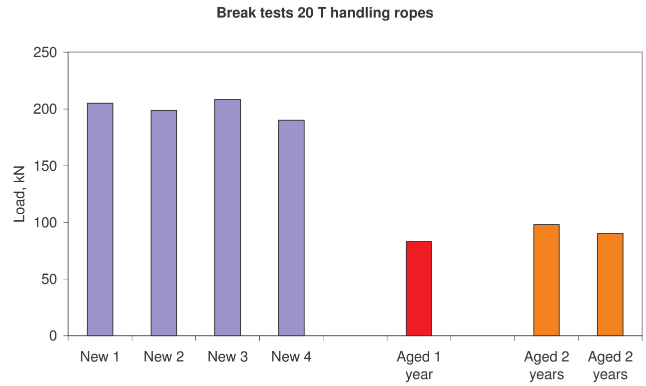


Figure 15. Strength retention, handling ropes

It is apparent that there is a significant drop in strength, even after one year in service, but this drop then stabilizes after two years. Assembled yarn samples were also taken from a larger, 50 ton break load rope used for coring for 7 years, and from tensile break tests on these again a drop of around 50% of rope break load was estimated.

Inspection of the rope core indicated extensive abrasion of the outer surface of the strands, while the heart of the strands appeared to be intact. Figure 16 shows the strand appearance compared to that of a new rope.

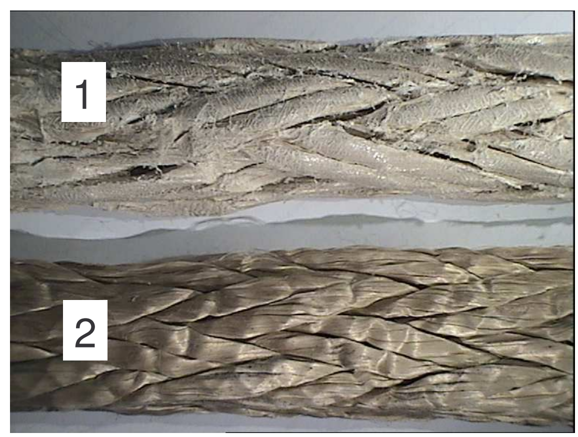


Figure 16. New and used rope after 7 years service, appearance with cover removed.

The excellent performance of aramid instrumentation lines in sea water is in sharp contrast with these results, which suggests that abrasion in sea water may be the critical combination leading to degradation. A program to study this aspect was therefore initiated.

Bend over sheave fatigue testing of 20 ton break load ropes

A study of the bend over sheave behavior of the 20 ton break load handling rope has been performed on the machine shown in Figure 14. Tests were performed over a 80 cm diameter reinforced nylon sheave identical to that used on the oceanographic vessel. The sheave was loaded in compression to 20 Tons, resulting in a tensile load of 10 Tons in the rope. It was then cycled under displacement control with a sinusoidal displacement of amplitude ±60 cm with a loading cycle period of 40 seconds, until failure. Figure 17 shows a five minute segment of the loading cycle.

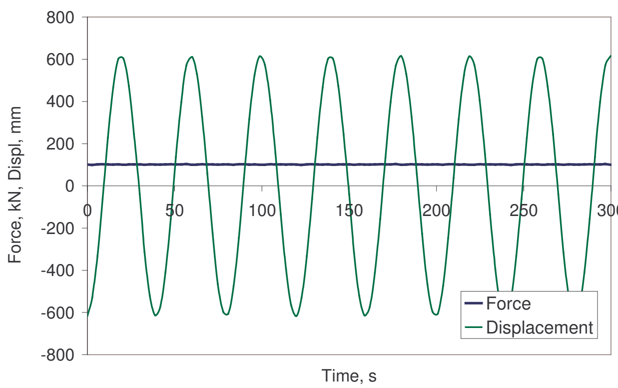
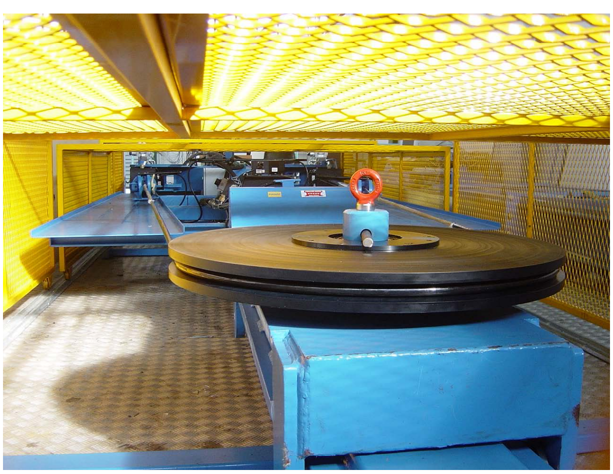


Figure 17. Load and displacement of handling rope sample during BOS tests

Figure 18 shows a rope on the test machine and results from 4 tests. The test on the third sample tested dry was stopped after 15000 cycles, residual tensile break load was then measured to be 16 Tons. Overall the cyclic behaviour of these ropes in the dry condition is good, though there is some variability in the results, and there was a small number of samples available. It should also be noted that this is a severe test, a small length of rope is continuously loaded over a sheave whereas in practice the loading will be distributed over a much longer length. A published study also indicates that dry *Technora T220* braided ropes show good BOS performance [21].



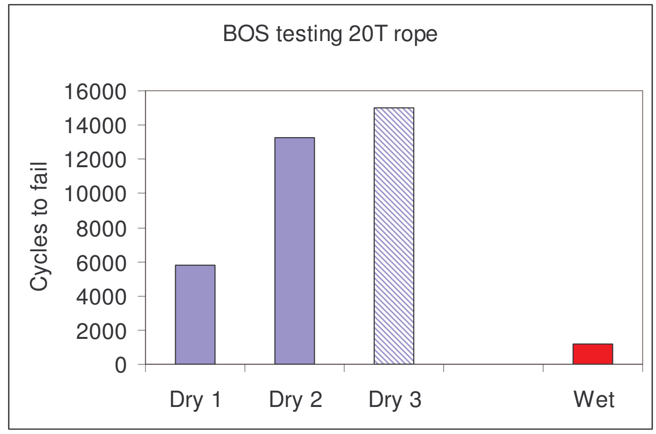


Figure 18. 20T handling rope on sheave and bend over sheave results.

The wet sample was immersed for 11 days at sea before testing, and sealed to keep it wet during the tests. The lifetime of this sample is significantly lower than the other results, suggesting that it is the combination of abrasion and water which leads to excessive wear. Examination under the microscope, Figure 19, also indicates an aspect after failure which is different to that of the samples tested dry, and similar to that seen on the samples aged in service (Figure 16) with a roughening of the outer core surface and the appearance of fibrils.

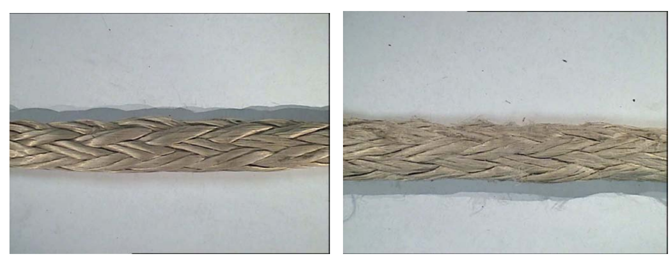


Figure 19. Sections taken from wet handling rope after BOS test
Left: region outside sheave, Right: loaded on sheave, zone near break.

These results raise a number of questions concerning the behaviour of these ropes in water. With a protective thermoplastic coating it might be assumed that the aramid core will not be exposed to water, but at high hydrostatic pressures diffusion through the coating may occur. Also at the rope ends the termination regions will not be perfectly watertight and will allow water entry. The similarity of the cores aged in service and tested wet suggests that this is the case but this needs to be confirmed.

IMPROVED MARINE FINISH

In order to improve long term durability the fiber suppliers *Teijin* have developed improved marine finishes. One example was supplied for the present study, reference *Technora T200W*. A study of the long term performance of this material is currently underway, but some preliminary tests have been completed and suggest that this formulation may significantly improve long term marine durability.

The first tests performed were yarn-on-yarn abrasion following the standard test procedure [22] in natural sea water. Figure 20 shows first results, together with test results for yarns removed from a 20 Ton handling rope. It is clear that the performance of the T200W yarn is significantly better than that of the yarn currently used, (note the logarithmic scale) suggesting that the improved finish may reduce wear in water.

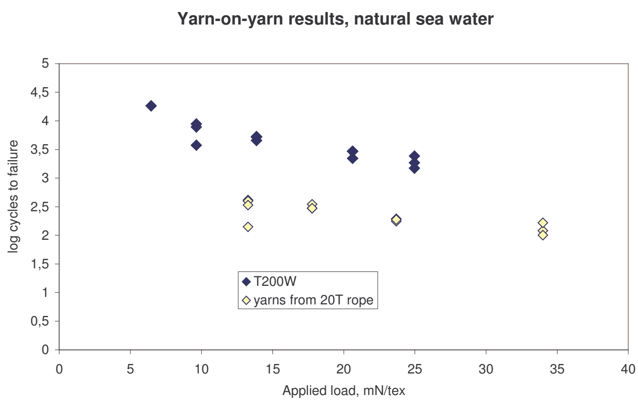


Figure 20. Yarn-on-yarn abrasion test results.

CONCLUSIONS

This paper presents first results from a study currently underway, aimed at understanding and predicting the lifetime of aramid fiber ropes employed in marine applications. The experience from oceanographic applications suggests that aramid fibers retain high residual properties after seawater exposure, degradation has only been noted after bending over sheaves. Improved marine finish on fibers may reduce this degradation, further work is needed to improve understanding of rope wear mechanisms in water.

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