Mechanical and Chemical Reliability Assessment of Silica Optical Fibres

Irina Severin, M. Caramihai, K. Chung, G. Tasca, T. Park

***Abstract*—**The current study has investigated the ageing phenomena of silica optical fibres in relation to water activity which might be accelerated when exposed to a supplementary energy, such as microwaves. A controlled stress by winding fibres onto accurate diameter mandrel was applied. Taking into account that normally a decrease in fibre strength is induced in time by chemical action of water, the effects of cumulative reagents such as: water, applied stress and supplementary energy (microwave) in some cases acted in the opposite manner. The microwave effect as a structural relaxation catalyst appears unexpected, even if the overall gain in fibre strength is not high, but the stress corrosion factor revealed significant increase in certain simulation conditions. .

***Keywords*—**optical fibres, mechanical testing, aging, microwave, structural relaxation.

I. INTRODUCTION

IN parallel to optical fibres' role as key components for telecommunication networks, the availability of inexpensive and high quality fibres has induced more and more applications in various areas such as sensing, remote chemical analysis, thermal measurements and thermal imaging, reflectometry, optical instrumentation, laser power delivery and fibre lasers. A number of applications relate to devices exposed to ionising radiations including nuclear plants, high-energy physics, plasmas and medical devices meaning certain harsh operational conditions. They could expand very significantly when technological progress leads to disposable fibres at a reduced cost.

In this respect, reliability issues have been studied carefully and much work has been achieved on this matter. The common requirement is that the fibre lifetime should be larger than the expected system operation time. Even if telecommunication networks have not faced yet major problems in relation to fibre failure, certain aspects of fibre aging are not fully understood, as cited by other authors, too [1].

The reliability issue remains more than ever a topical question for several reasons. On one hand, the impressive increase of the bit rate is accompanied by a power increase supported by the fibre cladding and might generate catastrophic failure phenomena as well as engender damage of the fibre ends or losses in the connectors. On the other hand, current models accuracy for lifetime prediction is questionable, these models including humidity, applied stress and temperature as major aging factors. Optical fibres reliability depends on various parameters that have been identified, such as: time, temperature, applied stress, initial fibre strength and environmental corrosion [2] – [6].

When studying optical fibres reliability, one should separate the case of the fatigue static behaviour where fibres are subjected to a permanent strain, e.g. bended fibres, and the dynamic fatigue corresponding to an unexpected tensile stress arising from environmental changes. Even if fibers are often replaced before their expected lifetime in order to comply with the new specifications of the network, failure mechanism involves surface phenomena, raising fundamental questions [7].

Literature reveals authors work on stress corrosion state-of-art, ***n*** calculus in dynamic, respectively static fatigue testing, reporting ***n*** dynamic fatigue value usually around 21-27 (other similar reference n=24), in case of silica optical fibre normally stored in laboratory for at least few months [8]. Other reference revealed a decrease of ***n*** in the case of long-term stored fibres [9], but based on our observations ***n*** stress corrosion factor of fresh manufactured optical fibres initially increased from 16-18 (immediately after manufacturing) to around 24 (after 6 months of storage in laboratory conditions), to decrease further for very long storage durations and certainly depending of the storage humidity fluctuations.

Different mechanisms have been proposed for ***n*** increase as static stress decreases. Silica bonds deform more by stretching rather then twisting, however, after some time, these bonds start twisting at the expense of the stretched already present in the bonds; this causes relaxation in the silica network. Optical fibre lifetime predictions revealed that fibre resists quite well in local loop environment (≤ 10 mm), coating degradation due to accelerated aging being not relevant in out-of-plant environment, ***n*** reported at 29 [10].

Once the aging simulation performed, optical fibre samples are tested [11], [12]. Lower breaking strength in tension tensile should be expected than in two point bending test, due to the longer gauge (0.2-0.5 m as compared with 1 cm) and the as-well phenomenon that longer fibres are weaker because of the greater number of flaws [13]. Increasing relative humidity during testing, breaking strength decreases and higher mechanical resistance is reported in dry than in wet

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environment [4], [13], [14]. Testing in wet environment is usually carried out by soaking samples for several minutes in this environment before starting the test.

Results treatment is then usually based on Weibull plots. Weibull distribution presents better description in bending than in tensile experiments [15], [16], possible argue through strength distribution strongly affected by the silica diameter variation (125±2.5μm), sufficient to account for observed deviations in fibre strength. It is reasonable to suppose that in tension the diameter variation (intervening to the square value) masks any size variation of surface flaws, to reword Kurkjian & Paek [17] proposal that fibre are essentially flaw free and that the diameter variations govern the strength. For a reasonable estimation using Weibull at least 50 samples are considered necessary. Other authors [18] proposed to consider for the strength calculus the rigidity and not only on the silica cladding diameter.

Our previous reliability studies [14], [19] concerning optical fibres aging for long periods (from several months up to 2 years), then shorter periods (from several days up to 3 months) allowed to develop the current work focused on the influence of a supplementary energy overlapping a constantly applied stress and the water corrosion factor. This supplementary energy was introduced by aging fibres, previously subjected to a controlled applied stress, to microwave in water environment (in liquid or gaseous phase).

II. EXPERIMENTAL PROCEDURE

A. Tested fibre

A commercial Alcatel 125/250 fibre, stored in uncontrolled laboratory environment for 6 years was used for testing. The silica cladding was of 125 μm in diameter, since the two-layer epoxi-acrylate polymer coating was of 250 μm in diameter.

Other series of testing was performed using Dätwyler Cables Singlemode fibre (SMF) Full-Spectrum SMF-28e+™ Optical Fiber E9/125/250 according ITU-T Rec. G. 652.D / IEC 60793-2-50 Type B1.3 – Corning fibre.

B. Tensile testing procedure

A tensile bench MTS Systems ADAMEL LHOMARGY (max. 100N) was used [7]. Tensile testing conditions were performed in a controlled environment with 17-19°C in temperature, 46-52% relative humidity and no more than 5 % variation during each series of the tensile tests.

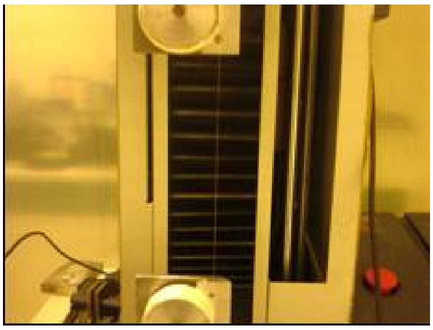
In order to be dynamically tensile tested, sample fibres were three tours rolled on the cylinder pulley having 65 mm in diameter. The pulleys are covered with a powerful double face adhesive; mechanical properties of the adhesive layer appeared important controlling factors, as mentioned by [13]. Despite the standard conditions, for economy and testing time reasons, sample testing free length was chosen to 200 mm (fig. 1).

Testing strain rate of 20, 100, 200, respectively 500 mm/min was chosen; some testing for the strain rate of 50 mm/min were performed, too. These rates were selected in order to correspond to 10, (25), 50, 100, respectively 250 %/min as compared to sample main length. The supplementary test at 50 mm/min was performed to check the ***n***-stress corrosion calculus or in case of some available samples subsequently the as-planned testing to validate the observed testing trends.

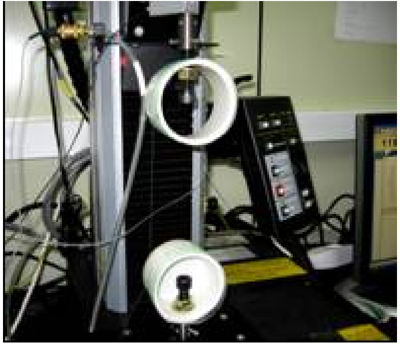
In the case of the reference fibre at least 30 samples were tested and Weibull plots were traced, then ***n***-stress corrosion factor was calculated. In the case of as-aged samples, less testing was performed, but at least series of 15-16 testing for each combination of aging duration and/or applied stress (respectively mandrel diameter).

For statistical reasons, in case of some anomalous values in result series of the fibre strength, they were not considered. Anyhow, one might noticed that no more than 10-15 % of the total series exhibited slightly out of range values than the overall tested series.

The second series of fibers was tested using an universal Testing Machine model Instron-5543, in a controlled environment with 30.7^{0}C temperature and 25% humidity and no more than 5% variation during each series of the tensile tests. In order to be dynamically tensile tested, sample fibers were three times rolled on the cylinder pulley having 87 mm in diameter. The pulleys were covered with a powerful double face adhesive. Sample testing free length was chosen to 200mm. Testing was performed using speeds of 20, 100, 200 and 500 mm/min respectively.



a)



b)

Fig 1. Tensile testing machine and griping system

In some tests tensile strain was measured and in some the axial strain with the use of a camera and sensor (Fig. 2).

Fig. 2. Tensile testing machine with attached video sensor

C. *Aging treatment*

Optical fibre was wound on alumina mandrel of 2.5, 3.2 and 3.8 mm in diameter (fig. 3). The extremities were clamped in two oblique cut practiced in the simple clamping rings, made of elastomer-rubber, mounted on the extremities of the alumina mandrels. A useful length for 3-4 tensile testing corresponds to an as-prepared mandrel.

Fig. 3. As-prepared wound mandrels for aging treatment

The as prepared mandrels (Fig. 3) were soaked in a container in cold deionised water for 5 minutes before aging (as other authors suggested, too [13]), then the container was introduced in a microwave oven (Daewoo) for several minutes ranging from 2, 5, 7 respectively 8 min. Fibre was subjected to an overlapped aging effect of microwave energy and hot water added to the significant bending strain applied through the as-wound mandrel.

The applied stress on the fibre depends on the mandrel diameter accordingly to the Mallinder and Proctor relation [14], [19], as follows:

$$\sigma = E_0 . \varepsilon \left(1 + \frac{\alpha' . \varepsilon}{2} \right) \tag{1}$$

where: σ : applied stress (GPa); E_0 : Young modulus (= 72 GPa for the silica) ; ε : relative deformation of the fibre; $\alpha' = \frac{3}{4}\alpha$; with α : constant of elasticity non-linearity (=6). The relative deformation of the fibre depends on the mandrel-calibrated diameter, as follows:

$$\varepsilon = \frac{d_{glass}}{\phi + d_{fibre}} \tag{2}$$

with ϕ the mandrel diameter (in µm); d_{glass} = 125 µm, the glass fibre diameter; d_{fibre} = 250 µm, the fibre diameter, including the double layer polymer coating. This leads, in the case of 125/250 telecommunication fiber, to the corresponding stresses of 2.743 and 2.31 GPa for the calibrated diameter mandrel of 3.2 and 3.8 mm, respectively.

Subsequently, as-wound mandrels were dried in uncontrolled laboratory environment (16-18°C, 35-40% RH) for one day; finally fibre samples were unwound just at the moment of the tensile test. Tensile testing was carried out as previously mentioned. Weibull plots allowed comparison in order to highlight the aging influence on fibre behaviour in the simulated conditions. Weibull plots recording in case of optical fibre dynamic testing was given elsewhere [7].

III. RESULTS

The reference Alcatel fibre Weibull plots for four different strain rates revealed coherent mono-modal Weibull plots accordingly the linear regression with $R^2 > 0.95$. Based on these results, a rather good ***n*** stress corrosion parameter was calculated having the value of ***n=30.5*** for the Alcatel reference fibre, with a regression coefficient of $R^2=0.97$.

The second fibre series, the Corning ones, lead to medium fracture load varying between 57 and 68.5 N (consistent with other authors results [14]) and a calculated ***n*** stress corrosion parameter of *16.5* with a regression coefficient of $R^2=0.98$ (fig. 4 and 5).

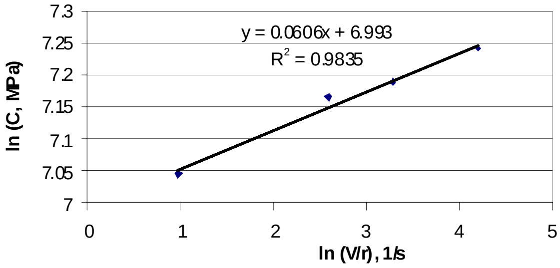


Fig. 4. n stress corrosion parameter calculus for the as-received fibre, dynamically tensile tested; in axes: failure stresses, C (in MPa) for different strain rates, V (mm/min), r optical fibre radius (mm)

For all tested strain rates, bent and aged fibre (3.2, respectively 3.8 mm mandrel diameter) presented an optimum of strength increase, as a result of the opposite effects of applied stress and microwave overlapping energy while aging.

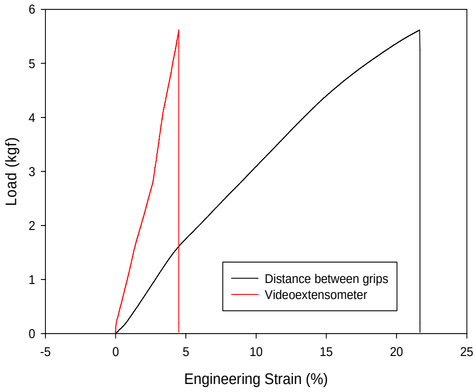


Fig. 5. Comparison of experimental results from the distance between two grips and that from video-extensometer. The crosshead speed of upper grip is 20 mm/min and the gauge length between two grips is 200 mm.

For the Alcatel fibre this optimum appeared for the duration of 2 minutes of aging in microwave (fig. 6). Higher applied stress (3.2 mm mandrel – 2.743 GPa) led to a little bit more effective increase - higher medium fibre strength as compared to similar fibre (reference, without microwave and other aging durations in microwave). Aging in water for few minutes determined globally a slightly strength decrease (as already noticed [20]), with a rather good mono-modal tendency for the strain rates of 100, 200 and 500 mm/min respectively, but bimodal one for the strain rate of 20 mm/min.

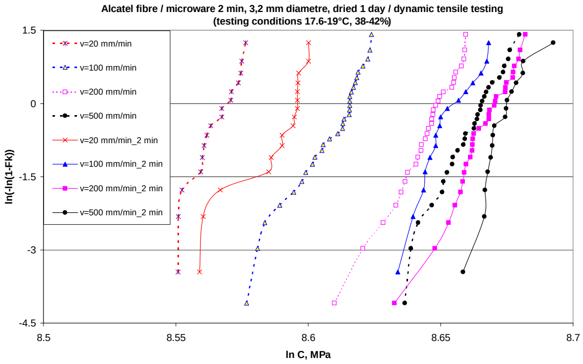


Fig. 6. Weibull plots of microwave-aged fibre as compared to the reference one in axes: failure stresses, C (in MPa) for different strain rates, v (mm/min), Fk cumulative fracture probability.

Excessive aging led in some cases to fiber fracture immediately before extracting from hot water at one of fixed extremities or along the aged length (sporadic behaviour), but anyhow no difference was noticed between testing results. Longer aging duration revealed a strength decrease, but, in these conditions, fiber don't loose drastically its' strength as compared with reference one. Same effects were noticed for lower applied stress, but with a lower gain in differences and strength increase (fig. 6).

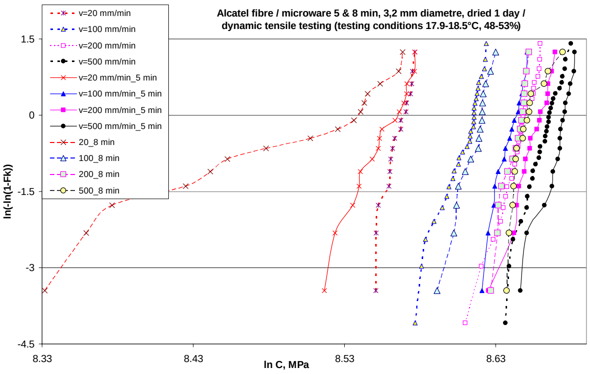


Fig. 7. Weibull plots of microwave-aged fibre as compared to the reference one in axes: failure stresses, C (in MPa) for different strain rates, v (mm/min), Fk cumulative fracture probability.

Summaryzing ***n*** stress corrosion evolution, for the optimum aging conditions (from the strength fibre point of view) – stressed on 3.2 mm mandrel and aged for 2 minutes in microwave, ***n*** stress corrosion factor appeared 17% higher (35.5 as compared to 30.5), to be noticed, with an acceptable, but a little bit larger dispersion (0.92 as compared to 0.972), too.

As seen in fig. 8, when prolonging aging (3.2 mm mandrel, 5 min in microwave), ***n*** stress corrosion factor decrease to 24 with a rather good regression coefficient (0.987). Continuing aging led to a more intensive decrease of ***n*** stress corrosion factor to 18.

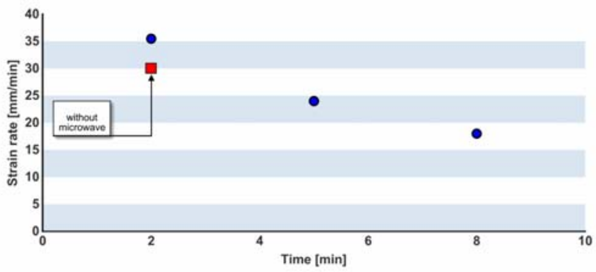


Fig. 8. Synthesis of **n** stress corrosion parameter evolution in different aging conditions with supplementary microwave energy.

V. DISCUSSION

As synthesis to our investigation, an enlarged scale of the fibre strength has allowed to notice that :

- generally the monomodal dispersion is present,
- aging in water for a similar duration slightly led to strength decrease,
- an optimum is noticed when aging in microwave for a 2 minutes duration,
- prolonging the aging duration revealed the strength decrease to lower values than the reference for 8 minutes,
- lower initially applied stress (2.31 GPa) through the mandrel diameter (3.8 mm) led to strength increase, but

the effect was less effective than in the case of applied stress of 2.743 GPa (3.2 mm).

The winding effect around alumina mandrel led to higher fibre strength than non-wound fibre. When the external fiber surface reaches the plastic strain, a local plasticity occurs exactly as for a rod submitted to a tensile test. When the bending test continues, the plasticity gradually progresses from the external surface to the inner zone. In general, the fibre becomes more plastic only when the stresses become higher than those observed in the tensile test.

The external fibre surface having undergone a plastic strain is lengthened more than the internal surface. After unwinding from the mandrel, the external fibre surface retracts and is subjected to compressive residual stresses. These compressive stresses are at the intreface between the fibre core and the polymer and are in opposite direction to the deformation induced by the dynamic tensile test. Thus the obtained fibre strength appears higher than that of non-wound fibre.

As stripped samples using methilen chloride presented no influence at cladding level in terms of curvature, fibres refound their expected liniaire shape.

The stress corrosion parameter for a fiber subjected to microwave treatment appears higher than that of a fiber whice underwent only one ageing process in hot water. Considering the relationship between the tensile velocity V and the stress intensity factor K_I , respectively the critical stress intensity factor K_{IC}, accordingly the ***n*** stress corrosion parameter one might notice that the effect of the microwave slows down the propagation of the fibre defects.

Taking into account that normally fibre strength decrease is induced in time by water chemical action, the effects of water, applied stress and microwave energy have acted on opposite manner. We already noticed that aging fibres under a controlled applied stress, in certain conditions, should improve the fibre strength [21].

The effect of microwave as structural relaxation catalyst was unexpected, even if the overall gain in fibre strength is not high, but the stress corrosion factor revealed significant increase in certain simulation conditions. One appreciates that the opposite mentioned effects have led to the noticed optimum for a given duration in microwave cumulated to the controlled applied stress. The results has appeared more relevant taking into account the stress scheme during aging, in relation with the as-wound fibre sample: tensile stress at the outer mandrel fibre section, respectively compression stress at the inner mandrel fibre section.

As already known, the micro-cracks smoothing due to the curing effect of water molecules on silica resulted in stress intensity factor decrease and thus, in fibre failure strength increase. But the smoothing effect might not be the only responsible for strength increase in the given conditions.

The evolution of fibre strength versus aging duration under controlled applied stress and supplementary energy (microwave) should be explained through the changes at fibre-polymer interface due to the structural relaxation. A layer of hydrated silica is likely to be formed at cladding fibre surface. This layer inhibited, in some extend, the micro-surface flaws. This vitreous hydrated phase may relax under stress at room temperature, which partly compensates the external applied stress through static fatigue (as-wound on mandrel). Our study is complementary to that of Tomozawa & Hepburn [22] detailing different failure mechanisms of silica, including surface structural relaxation.

The noticed behaviour in certain conditions may present some practical consequences suggesting that in wet environments, in some particular cases (temperature, exposure duration, applied stress, supplementary energy such as microwave), the as-aged fibre strength has slightly exceeded the expected one, respectively the non-aged fibre strength, with a more significant increase of the ***n***-stress corrosion factor. The combination of factors leading to this significant increase was considered as optimum aging conditions.

VI. CONCLUSIONS

The current study has revealed that failure mechanism of as-aged fibres involves surface phenomena, in relation to water activity that might be accelerated when overlapping a supplementary energy, as the microwave one.

The evolution of fibre strength versus aging duration under controlled applied stress and overlapped supplementary energy, in the investigated case, microwave energy should be explained through the changes at fibre-polymer interface due to the structural relaxation. One might anticipate that controlling the relaxation phenomenon could lead to the increase of the fibre strength and its' reliability when subjected to static and dynamic fatigue, too, as the case in practical environments.

The current and other previous studies allowed us to conclude that an optimum combination of controlled stress and aging conditions accordingly the fibre type should be identified based on multiple experimental simulations.

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