Optical fibre sensors for monitoring ageing of materials : Application to polyethylene and metallic structures

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Two examples of in situ monitoring of ageing of materials with optical fibres are shown in this paper: thermal degradation of polyethylene and corrosion in aeronautic structures (aluminium) with an optical fibre corrosion sensor (OFCS).

The thermal degradation of polyethylene is monitored by inclusion of an uncladded optical fibre in the polymer. Through modelling of the angle distribution of the normalized light power transmitted with geometric optics, the refractive index of the polymer is determined at different temperatures ($n=1.4594 - i0.0007$ at 28°C and $n=1.5162 - i0.0007$ at 120°C). The variation of the refractive index is related to a variation of crystallinity of the polymer. The kinetics of degradation was directly followed at different temperatures.

The OFCS is tested by two methods: optical and electrochemical methods for different aluminum thickness deposited on the sensitive part of the optical fibre, in different acid concentration. The mechanisms of corrosion (uniform or in preferential places) can be deduced from the optical signal. The sensitivity of the OFCS is good for the low thickness. Corrosion rates for aluminum and copper are quite different, the effect of oxide layer on aluminum is important and induces corrosion in several stages.

Keywords: Optical fibre, Evanescent wave optical sensor, Refractive index, Crystallinity, Kinetics of corrosion, Aluminum film, Potentiometric measurements

I. INTRODUCTION

Among all the sensors (dielectric, ultrasonic...) to actively follow the development process or the decomposition of polymers, the optical fibres have many advantages. They are at the same time the sensitive element (low dimension) that can be included in the structure itself and the information vehicle, which confers a very short response time to them and no perturbation from electromagnetic environment. They are very cheap and core material can fit with the optical properties of the polymer. Work showed that it was possible to integrate optical fibres in polymers or stratified

composites without significant modification of the mechanical properties.¹⁻⁴

It remains to be known if it can bring relevant information as for the physical or chemical follow-up and or chemical of the mechanisms related to the degradation reaction of polymers. One can thus imagine only one optical fibre able to control the development process actively to provide information on the mechanical properties of the structure in service and, why not, follow the chemical ageing of polymer.

The sensors using optical fibre for the follow-up of polymerization of the thermohardening resins

are numerous. The publications⁵⁻⁹ generally make state since 1990 of various techniques, based either on the measure of the absorption of the evanescent wave (in near infra-red spectrometry near infra-red), or on the measure of the resin refraction index. The experimental approach which was selected for this study uses this last solution. It is however original in its form because it is based on measurement, in monochromatic light, of the angular distribution of the power transmitted by the fibre-sensor. This technique results from the experimental set-up built in our laboratory as regards to sensors optical fibre for the detection of gas¹⁰ and biochemical species.¹¹

In the design of aircraft, the economic life is assumed to be 20 years.¹² The two primary damage mechanisms that limit the life of metallic structure are fatigue and corrosion. Corrosion has been found a problem that increases with age, particularly when the original design lifetime is exceeded. Thus, a strong demand of in situ monitoring of corrosion exists, hidden sensors included in structures which could give an early indication of the corrosion without significant disassembly, could reduce cost of maintenance.

Many techniques for studying and detecting aircraft structures corrosion have been developed. A simple vibration-based method of damage detection for monitoring ageing structures has been developed.¹³ Hidden corrosion is detected in aircraft aluminum structures using a noncontact laser based ultrasonic technique.¹⁴ The microsampling technique has been shown to be an accurate method for obtaining samples from occluded regions on aging aircraft.¹⁵ Electrochemical impedance spectroscopy (EIS) has been used to characterize corrosion phenomenon.^{16,17} The growth behaviour of fatigue cracks initiated and comprehensive corrosion fatigue model has been investigated due to pitting corrosion fatigue in aging aircraft structures.¹⁸⁻²⁰ Nature and cause of corrosion are investigated in the aircraft radar antenna waveguide.²¹

Optical fiber corrosion sensors have been analysed and tested utilising the sensitivity of

surface plasmon resonance (SPR) and Bragg reflection influenced by SPR.^{22,23} Metallised optical fibres have been developed in order to follow the corrosion in aeronautic structures.²⁴ In 'Ref. 25' the research on optical fibre corrosion sensors (OFCS) fabricated by electroplating an Fe-C alloy film onto an optical fibre core within the sensing region is reported. In our laboratory, was demonstrated the feasibility of an optical fibre corrosion sensors when the sensing region of the optical fibre is metallised with a chemical nickel-phosphorus deposit²⁶ and with a copper film deposited through electroless method.²⁷ The last paper shows that the degradation of the copper surface by corrosion is uniform, in acid solution at low concentration and the optical sensor manufactured is sensitive in high acid concentration, when corrosion takes place through pits and cracks.

In this work, our goal is to develop an optical fibre corrosion sensors (OFCS), fabricated by thermal deposition of an aluminum film onto an optical fibre core within the sensing region, in order to follow corrosion in aeronautic structures.

II. EVANESCENT WAVE OPTICAL FIBRE SENSOR FOR THE IN-SITU MONITORING OF THERMAL DEGRADATION OF POLYETHYLENE

II-A. Experimental set-up

For the experiment, a fibre was manufactured at the Institute of Radio Engineering and Electronics with a high refractive index core ($n = 1.62$) prepared from soft optical glass (F2 glass). With this fibre, Polyethylene (PE) can be used as optical cladding. The fibre core has a diameter of $300 \pm 5 \mu\text{m}$ and it is coated with a siloxane cladding (Sylgard 184, USA, $n = 1.41$) with a thickness of $50 \mu\text{m}$ (the fibre numerical aperture is 0.8). Fig.1 presents the principle of operation of the optical fibre sensor. The input and output faces are polished perpendicularly to the fibre axis. The original siloxane cladding was removed from the central part of the fibre on a length of 2cm, with a scalpel and then the core was cleaned with acetone

Fig. 1. Operation principle of the optical fibresenso.

and ethanol. This stripped region is therefore the sensing element of the sensor. The others parts are insensitive to the external medium, since the siloxane cladding refractive index is lower than the core refractive index and the evanescent wave penetration depth ($1\ \mu\text{m}$) is lower than the thickness of the cladding ($50\ \mu\text{m}$). The fibre was placed between a laser diode (ILEE, LD A02, 2 mW, 670 nm) and a silicon photodiode (TELEFUNKEN S135P) with a surface of $16\ \text{mm}^2$. The laser source was mounted on a rotary stage. Then, the transmitted light power was measured according to the incident angle. A computer acquired the photometer signal.

The polyethylmene is put in the heated mould made of three plates (two hot plates and one moulding plate) and crossed by the uncladded part of the optical fibre and a thermocouple was placed in the polymer.

II-B. Results and discussions

II-B-a. Test of sensitivity

Initially, the sensor sensitivity is determined in an aniline-ethanol mixture. This mixture had several advantages:

- chemical inertia between ethanol and aniline,
- the mixture of aniline-ethanol offered a broad range of sensitivity between 1.58 and 1.36 of refraction index.

- the two liquids chosen, aniline and ethanol, are transparent, one can then measure their refractive index with a Abbe refractometer. First of all, one carried out with an Abbe refractometer the measurements of refractive index for different mixtures of aniline-ethanol (Fig. 2).

Fig. 2. Measurement of the refractive index of ethanol-aniline mixtures with a Abbe refractometer.

Then, different mixtures were used to perform the sensitivity tests on the optical fibre sensor. After cleaning, the sensitive part of the fibre was introduced into a tank containing the aniline-ethanol mixture. The response of the optical fibre sensor was evaluated in the range of refractive

index reached during polyethylene degradation. The experimental values of normalized light power transmitted P versus the injection angle α , for the uncladded fibre inserted in the polyethylene, are presented in Fig. 3. The modelling of the experimental data is performed using geometrical optics as previously described,²⁸ taking into account skew light rays. An energy loss is introduced at each reflection at the core-cladding interface by an extinction coefficient (imaginary part of the refractive index). The value of the refractive index of the polyethylene at 28°C is found to be $1.4594 - i 0.0007$ and the value of the refractive index of the polyethylene heated for more than ten hours at 120°C is found to be $1.5162 - i 0.0007$.

The objective of this preliminary test was to determine the best incidence angle for the monitoring of the degradation kinetics. Fig. 4 shows the evolution of the angular distribution when the sensor is immersed in a ethanol-aniline mixture with various refractive index. The sensor presents a good response to the variation of the refractive index. It is impossible to quickly measure the angular distribution versus the time of

polymerization. The monitoring of the polymer degradation was performed at a fixed angle. The incidence angle for a higher sensitivity to the variations of refractive index is not constant. It depends, indeed, of the medium refractive index to be tested. From 1.45 to 1.52, the incidence angle of a higher sensitivity varies between 15° and 25°. The average value of 20° was then selected.

II-B-b. Monitoring of PE degradation

In these experiments the sensing region of the fibre was placed in the heated polymer. Fig. 5a shows the recording of the response of the optical sensor for a temperature of 100°C. In a first phase, optical signal remains invariant. After 12 hours, the signal of the photometer increases gradually and then reaches a limiting value after 5 hours. After 17 hours, no significant variation of the signal is detected.

Three similar experiments were carried out at various degradation temperatures: 110°C, 120°C and 160°C (Fig. 5b, 5c and 5d respectively). At 110°C, one observes a behavior of degradation similar to that observed at 100°C. For the higher

Fig. 3. Angular distribution of the normalised light power transmitted by the optical fibre sensor introduced into polyethylene.

Fig. 4. Response of the sensor immersed in various aniline-ethanol mixtures.

temperatures, one sees that the variation of the signal is abrupt at 120°C. The signal is stabilized after about 100 minutes. At temperatures higher than the melting point, 160°C, the variation of the signal is almost instantaneous and stabilizes after a few minutes.

The variations of the optical signal is correlated to a variation of the refractive index of the polymer during heating as shown in § II-B-a. X-rays diffraction measurements (cf. Fig. 6) show that crystallinity increases with the heating temperature. It is however difficult to connect this parameter to the permittivity of polymer and to its refractive index.

III. OPTICAL FIBRE CORROSION SENSOR FOR AIRCRAFT APPLICATIONS:

III-A. Experimental

III-A-a. Principle

The principle of an OFCS is presented in Fig. 7, the metallised part of the optical fibre is immersed in an acidic solution. When the corrosion of aluminum coating takes place, parts of the metallic cladding around the core is removed and constitutes the corrosion products, the core coming directly in contact with acid solution. Hence, attenuation of the higher guided modes should decrease significantly and this phenomenon can be

detected by optical measurement as shown in Fig. 8. A change in the concentration of corrosion products in acidic solution can be determined by a simple measurement of the electrochemical potential.

So, we can couple these two methods by correlating the parameters describing the optical signal (of $P(\alpha)$ curves, angle at the inflection point of $P(\alpha)$ curves, full width at half maximum) and those describing the electrochemical corrosion process (electrochemical potential as a function of ionic concentration).

III-A-b. Set-up

In our experiments, the fibre used were PCS fibres from Quartz and Silice with a core diameter of 400 μm . The numerical aperture of this PCS fibre is $\sin(\alpha_{\text{NA}}) = 0.48$ with $\alpha_{\text{NA}} = 28.5^\circ$. When the incidence α angle of the light, which is defined as the angle formed by the incidental ray and the normal to the fibre entrance surface, is less than the value of α_{NA} , the total internal reflection occurs without any light reflecting out of the core.

The original silicone cladding was removed mechanically from the central part of the fibre on 5 cm length. Then, the core was cleaned with acetone. Stripped part was therefore the sensing part of the

sensor and a film of metal, aluminum or copper in our case, was deposited on it (Fig. 9). The total internal reflection boundary disappears when fibre core is metallized, and the light propagation in the

core of this fibre is mostly absorbed by the metal cladding and fades out along the length of this new fibre cladding.

Fig 5. In situ monitoring of PE thermal degradation at various temperatures: incidence angle 20° .

Fig. 6. XRD pattern of PE at different degradation temperatures.

Fig. 7. Principle of the optical and electrochemical measurements.

Fig. 8. Angular distribution of the light power transmitted by the PCS400 fibre.

Fig. 9. Aluminium surface deposited.

So, the metallised part of the fibre was maintained in contact with an agitated aqueous solution with a fixed concentration of nitric acid. The fibre was placed between a laser diode (ILEE, LD A02, 2 mW, 670 nm) and a silicon photodiode TELEFUNKEN S135P) with a surface of 16 mm² (cf. Fig. 10).

The laser source was mounted at a fixed incidence angle. So, the transmitted light power was measured at this sensitive incidence angle. In parallel, two electrodes (calomel reference electrode and a platinum electrode) were placed in the aqueous solution in contact to detect the change in the electrochemical potential due to the variation of Al³⁺ ionic concentration. Optical and electrochemical data were acquired with a computer.

III-B. Deposition techniques:

III-B-a. Electroless method (for copper)

In the electroless technique used for copper deposition in our previous work, the metallisation was performed following a procedure in seven steps:²⁸

- 1- Mechanical uncladding of optical fibre for about 5 cm.

- 2- Sulfo-chromic treatment, and washing with deionised water.
- 3- Immersion in a 1 M H_2SO_4 solution, at room temperature for 12 min.
- 4- Immersion in a colloidal solution Pd/Sn for 20 min.
- 5- Immersion in 1N hydrochloric acid solution for few minutes and then rinsed in deionised water.
- 6- Rinsing in deionised water.
- 7- The electroless metal plating was performed by immersion of the sensitive part of the fibre in a saturate metal solution.

III-B-b. Thermal evaporation method (for aluminium deposition)

Once the optical fibre was uncladded in central part, the uncladded zone was cleaned perfectly with successive acetone, ethanol and deionised water solutions. The fibre was then introduced into a vacuum chamber for thermal evaporation (Fig. 11). A mechanical system allowed the rotation of the fibre during the phase of deposition to obtain homogeneous film. The configuration of the evaporator limited the overall length of the fibre to 25 cm and the length of the sensitive zone to 8 cm. The aluminum evaporation was carried out under a pressure of about 10^{-7} torr, the fibre was placed just opposite the crucible filled with metal (metal

powder or bars), the crucible was heated till more than 1000°C . When evaporation of the metal occurs, it laid on the exposed uncladded zone of the fibre and on the interior walls of vacuum chamber. Aluminum thickness deposited was determined by the aluminum quantity evaporated and by the heating the crucible. Checks of the thickness were carried out on glass plates introduced and put near the uncladded zone showed a very good reproducibility of the deposits and a very good appreciation of their thickness. Tests were carried out using a profilometer (Dektak).

III-C. Results and discussion

III-C-a. Aluminum corrosion

Results and observations

The tests of corrosion were carried out on optical fibre corrosion sensors (OFCS) with different aluminum thicknesses deposited and in different concentrations of nitric acid. Sensitive incidence angle was fixed at 12° .

For an aluminum deposit of about $5\mu\text{m}$ thickness (Fig. 12 a) b)), the stage of saturation is obtained after long time for the weak acidic concentrations compared to that when the OFCS is immersed in high concentration of nitric acid solution.

Fig. 10. Experimental set-up.

Fig. 11. Thermal evaporator.

The optical stage of saturation is reached after about six hours in 10^{-2} N nitric acid concentration (cf. Fig. 12 b)) and not exceed one hour in 1N of nitric acid solution (cf. Fig. 12 a)).

The same behaviour is observed for an aluminum deposit of $2\mu\text{m}$ thickness (cf. Fig. 13 a) b)). Nevertheless, degradation of OFCS for a $5\mu\text{m}$ aluminum deposit is slow compared to that of the sensor with a $2\mu\text{m}$ aluminum deposit, in the same nitric acid concentration: It is almost two times longer in a 10^{-2} N nitric acid solution.

In order to correlate the two detection processes for monitoring corrosion, optical and electrochemical detection, the parameter time was omitted and optical detection was directly presented as a function of the percentage of corroded aluminum determined by electrochemical detection (cf. Fig. 14 and 15).

For a thickness of about $5\mu\text{m}$, in Fig. 14 is reported the variation of the optical signal according to the percentage of surface corroded for different nitric acid concentrations. For 1N nitric acid concentration, the curve can be divided into two zones: In the first zone, one observes progressive increase in optical signal according to

percentage of surface of aluminum corroded. In the second zone, the stabilization of the optical signal is observed from 50% of corroded surface, limit of corrosion sensor sensitivity. For the low nitric acid concentrations (10^{-1} N and 10^{-2} N), one observes three stages on which the optical signal is stabilized. Two first stages followed by a sudden change of signal and a third stage of saturation:

- The first stage when the optical signal is invariant: uniform corrosion layer by layer of the metal surface. The limit of the stage is at about 50% of surface corroded in 10-1N and exceeds the 60% in a concentration of 10^{-2} N. This stage is followed with a sudden increase in the optical signal corresponding to a removing of large size pieces of remaining thin metallic layer.
- Stabilization again of the optical signal: uniform corrosion by the metal surface of the remaining metallic layer. The limit of the stage for the two concentrations is located at 90%. The second stage is followed by a sharp increase in the optical signal: separation of large size pieces of the metal layer.
- The last stage of stabilization or saturation: limit of the sensor detection.

In the same way for an aluminum thickness of about 2 μm , in Fig. 15 is reported the variation of optical signal according to percentage of surface corroded for the different nitric acid concentrations. For the high nitric acid concentrations (1N and 10^{-1}N), one observes a progressive increase in signal followed by stabilization: corrosion in preferential places.

For low nitric acid concentration (10^{-2}N), one observes a light increase in optical signal which

extends up to 90% of aluminum surface corroded: soft corrosion, layer by layer.

The 10% of remaining metallic layer is very thin. When corrosion is going on, large pieces of metallic layer are removed and the fibre core comes then in contact with the acid solution and the numerical aperture is suddenly increased leading to a high increase of the optical signal.

Fig. 12. Evolution of the aluminum layer corrosion of 5 μm deposited on an optical fibre.
a) high acid concentration (1N) b) low acid concentration (10^{-2}N)

Fig. 13. Evolution of the aluminum layer corrosion of 2 μm deposited on an optical fibre.
a) 1N b) 10^{-2}N

Fig. 14. Variation of the optical signal along the aluminum layers corrosion process of 5 μ m.

Fig. 15. Variation of the optical signal along the aluminum layers corrosion process of 2 μ m.

Interpretation of the results

The results for aluminum corrosion have shown that the growth and propagation of corrosion pits is associated with the formation and rupture of hydrogen blisters as shown in Fig. 16. Such blisters undergo several stages of development.²⁹⁻³² These

are (i) formation of a blister beneath the oxide film due to electrochemical reactions occurring at the oxide/metal interface, as described above, (ii) growth of the intact blister due to continuing electrochemical reactions and concomitant changes

Fig. 16. Scanning electron micrograph of the top view of oxide blisters on aluminum surface alloys.²⁹

in the local electrolyte within the blister, and (iii) eventual rupture of the blister due to the build-up of hydrogen pressure within the blister. Once pitting has been initiated, changes in the occluded cell electrolyte occur as in the case of crevice corrosion. Locally dissolved aluminum ions hydrolyze to produce an acidic environment, and nitrate ions then migrate into the occluded cell to maintain charge neutrality. Propagation of the corrosion pit is thus due to anodic dissolution in the occluded pit environment (low pH, high nitrate ion concentration, high concentration of metal cations).

For high acid concentration, concentrated acid attacks on preferential places occur. Therefore, the hydrogen pressure is sufficient to cause the blister cracks. So, pitting propagates in these damaged places until fiber core is reached.

For low concentrations, the aluminum corrosion is done in an homogeneous way. The hydrogen pressure is not sufficient to cause blister cracks. Therefore, the blisters widen and coalesce with the adjacent ones. So, blister surfaces remain significant and when hydrogen pressure is sufficient blister crack, which gives a significant corroded surface. The corrosion thorough in uniform way until fiber core. Therefore, the surface of contact solution/core will be significant, which explains the sudden increase of the optical signal shown previously in low nitric acid concentration.

For low acid concentrations and thickness of about 5μm of aluminum deposited, one notes that there are several stages of saturations when the optical signal is invariant with metal corrosion. But,

for low thicknesses of about 2μm, the sensitivity of the OFCS is good; the sensor detects the least variation due to the corrosion phenomenon.

III-C-b. Comparaision of corrosion of aluminum and copper

On Fig. 17 and 18, one reports the comparison of corrosion tests made on an OFCS elaborated by electroless of copper and an OFCS elaborated by thermal evaporation of aluminum in 10^{-1}N and 10^{-2}N nitric acid solutions respectively.

For copper one observes that there is a continuous increase in signal until reaching the limit of sensitivity of the OFCS where the optical signal is invariant. There is progressive increase for a acid concentration of about 10^{-1}N and a sudden increase in 10^{-2}N acid solution.

But in the case of aluminum corrosion, one observes three stages of corrosion in 10^{-1}N and 10^{-2}N acid solution. Two first stages followed by a sudden change of signal and a third stage of saturation.

Corrosion rates for aluminum and copper are quite different. The effect of oxide layer on aluminum is important. The shape of the curves optical signal versus electrochemical signal allows to translate these different corrosion mechanisms: sudden step in the case of aluminum and continuous curves in the case of copper.

Fig. 17. Behaviour of OFCS elaborated by aluminum thermal evaporation and chemical copper electroless for low acid concentration. ($\approx 5\mu\text{m}$ thickness)

Fig. 18. Behaviour of OFCS elaborated by aluminum thermal evaporation and chemical copper electroless for high acid concentration. ($\approx 5\mu\text{m}$ thickness)

IV. CONCLUSION

Two examples of in situ monitoring of ageing of materials with optical fibres are shown in this paper: thermal degradation of polyethylene and corrosion in aeronautic structures (aluminium) with an optical fibre corrosion sensor (OFCS). A differential mode measurements between a sensitive fibre and a non sensitive fibre would allows cancelling of the effects of outer parameters such as temperature, humidity and instability of the laser source. The interest of the use of the optical fibre sensors should be moreover emphasized by using refractometry in order to be able to localize the ageing phenomenon.

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