## STUDY ON THE NEOPRENE DEGRADATION UNDER COMBINATION EFFECT OF OVEN AGEING AND FATIGUE

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#### Résumé

La prévision de la durée de vie du néoprène constitue un enjeu industriel majeur pour l'ensemble des éléments mécaniques fabriqués à base de ce matériau comme les flexibles et les courroies industriels et automobiles. L'utilisation de ce matériau a montré qu'il se dégradait encore plus vite sous l'effet combiné de la température et des sollicitations cycliques. Dans le but de caractériser la relation entre le vieillissement et la contrainte d'endurance nous avons entrepris des essais de fatigue en traction alternée sur un ensemble d'échantillons de néoprène neufs et vieillis à 100°C dans une étuve universelle ventilée pendant 48h et 96h. Les résultats expérimentaux montrent, d'une part, que les courbes de Wöhler pour les trois échantillons ont la même allure et que, d'autre part, La contrainte d'endurance chute brutalement entre l'échantillon neuf et l'échantillon vieillis à 100°C pendant 48h et elle se stabilise ensuite entre ce dernier et l'échantillon vieillis à 100°C pendant 96h.

Mots clés : Néoprène, fatigue, étuve, endurance

#### Abstract

The forecasting of the neoprene lifetime constitutes a major industrial stake for the set of the mechanical manufactured elements with this material like the flexible shaft and automotive belts. The use of this material in these cases, besides, shown that this material deteriorated again more quickly under the combination effect of temperature and fatigue. To characterize this, we carried fatigue tests in alternate tension on a set of new neoprene specimens and ageing ones during 48h and 96h at 100°C into a universal ventilated steam room. The experimental results show, that the (S,N) curves for the three sets of specimens have the same shape and that the fatigue strength brutally falls between the new sample and the aged ones, indicating a loss of fatigue characteristics of this material under accelerated ageing.

Keys words : Neoprene, fatigue, oven ageing, fatigue strength

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### ملخص

ا**لكلمات المفتاحية**: العياء ، البوليكلوروبرين ، وهـلر

As a result mainly of its flexibility, wear and chemical resistance, neoprene is utilized in many applications, the mechanical manufactured elements. Neoprene is used for such things as flexible shaft and automotive belts. Unfortunately, neoprene performance versus time degrades due to ageing, and the extent of this degradation is very difficult to predict [1].

This is because the rate at which neoprene degrades depends on many factors such as the operating temperature, chemical environment, loading conditions [1]. Ageing results in a loss of flexibility, abrasion resistance and elasticity. In some abrasive or erosive applications, this time scale of degrading properties is not a problem as the neoprene will be worn away before any serious loss of properties occurs. In some cases this unpredictability of degradation rate can lead to catastrophic failure in service such as a conveyor belt breakage, which causes downtime and can be very dangerous [2].

At present inspection techniques for neoprene conditions rely upon observing the subsequent ageing effects which in many cases can be too late to prevent failure. Neoprene consists of long, flexible polymer chains inter-connected via physical or chemical cross-links. The cross-link density for most applications must be sufficient to give the neoprene mechanical integrity so that it can bear loads and recover deformation. However, the cross-link density should be not so high that the polymer chains are immobilized, which would lead to a hard, brittle neoprene.

The optimum cross-linking between polymer chains gives a neoprene good flexibility and elasticity leading to high wear resistance and resilience to flexing - two of the most important features of neoprene. Neoprene ageing is a process of chemical reactions occurring between the neoprene and its environment. These reactions may change the polymer chain length, cross-link density and/ or chemical structure [3].

Fatigue life prediction in metallic materials has been largely investigated over the past decades and is still of major issue. In comparison to metallic materials and despite their growing use in wide range of industrial applications, fatigue life prediction in neoprene has been few investigated. For several decades fatigue life prediction has played a major role in structure design. As metallic components, polymeric components subjected to cyclic loading will fail by fatigue and appropriate fatigue life criteria are needed to prevent fracture in service [4, 5].

The purposes of this paper are: (1) to characterize the degradation neoprene due to accelerated ageing in ventilated steam room under static tension and tension-compression tests and (2) to establish from those observations a characteristic function of fatigue strength versus ageing time. To strike these objectives we undertake tension and fatigue tests on ageing neoprene specimen in ventilated steam room during 48 and 96 hours at 100°C and on new ones.

#### **1. EXPERIMENTAL PROCEDURE**

#### 1.1. Material

Neoprene (polychloroprene) is obtained in our laboratory from a mixture of acetylene in a concentrated solution of cupreous chlorides and ammonia chlorides (1) :

$$CH = CH \xrightarrow{Cupreous chlorides} CH = C - CH = CH_2 (1)$$

$$Monovinyla cetylene$$
(plus divinélacetylene)

We eliminate afterward the divinylaceylene by separation and we make steal the monovinylacétylène in a containing solution of cupreous chlorides and ammonia chlorides (2) :.

$$CH \equiv C - CH = CH_2 + HCI \xrightarrow{Cupreous chlorides} CH_2 = C - CH = CH_2 (2)$$

$$Monovinyla cetylene chloroprene$$

01

The chloroprene can be polymerized with emulsion technique. Polymerization occur enough quickly in controlled conditions (3) :



# 1.2. Moulding of neoprene tension and fatigue specimens

In the mix and emulsion vat, we take a volume of neoprene dough maintained at 160°C and then we spread it in a mould with 36 cell tension specimens. Under the combined action of heat and pressure the neoprene dough, inside the cells, takes the shape of the specimens described in figure 1.



Figure 1 : Moulded specimen scheme

## 1.3. Accelerated ageing processing of tension and fatigue specimens in a ventilated steam room

In a ventilated steam room, the specimens are suspended with tongs and ventilated with 100°C forced warm air during 48 hours for the first batch and during 96 hours for the second one. It's important to know that aging during 100 hours in a ventilated steam room at 100°C represents 4 years in normal conditions of pressure, temperature and air humidity.

#### 1.4. Tension Tests

Tension tests are conducted at a room temperature on an IBERTEST computer-controlled tension machine on three sets of neoprene specimens, the new ones and the ageing ones. The specimens are fixed on the superior crossbar jaw and dragged by a hydraulic jack. Strain and load are measured with an extensometer and a 20 daN cell built-in with machine. Both are recorded by a computer.

#### 1.5. Fatigue tests

Fatigue tests are conducted at a room temperature on a LLYODS INSTRUMENTS LR 10K type computer controlled fatigue machine on three sets of neoprene specimens, the new ones and the ageing ones. The experiments consist in conducting tension-compression tests with previously fixed load and with recording the number of rupture cycle. Tests are performed with a sinusoidal waveform at a frequency of 12 Hz.

#### 2. EXPERIMENTAL RESULTS

#### 2.1. Ageing influence on mechanical properties

Figure 2 shows the strain-stress curves of the three sets of neoprene specimens used in our tests. The mechanical properties of these sets of specimens are presented in table 1.

 $\underline{\text{Table 1}}$  : Mechanical properties of the three sets of neoprene specimens

Mechanical properties Specimen	Young modulus E (MPa)	Yield stress Re (MPa)	Maximal stress Rm (MPa)
New specimen	14,77	2,90	4,68
Ageing specimens during 48 hours at 100 °C	8,98	2,30	3,60
Ageing specimens during 96 hours at 100 °C	8,04	2,26	3,24



Figure 2: Strain-stress curves of the three sets of neoprene specimens

A comparative study between the mechanical properties of the three sets of neoprene specimens is led while taking reference values as the mechanical properties of the new set of neoprene specimens (see table 2 and 3).

T <u>able 2</u> :	Comparison	table between	n mechanical	properties	of new
specimen	and ageing o	one during 48	hours at 100	°C	

Mechanical properties Specimen	Young modulus E (MPa)	Yield stress Re (MPa)	Maximal stress Rm (MPa)
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Ageing specimens during 48 hours at 100 °C	8,98	2,30	3,60
Mechanical property decrease between new specimen and aged one during 48 hours at 100°C in percen	39	21	23

Table 3 : Comparison table between mechanical properties of new specimen and ageing one during 96 hours at 100°C

Mechanical properties Specimen	Young modulus E (MPa)	Yield stress Re (MPa)	Maximal stress Rm (MPa)
New specimen	14,77	2,90	4,68
Ageing specimens during 96 hours at 100 °C	8,04	2,26	3,24
Mechanical property decrease between new specimen and aged one during 96 hours at 100°C in percen	45	22	31

The different results presented here over show neoprene flexibility and elasticity loss versus time. The Young modulus is the more degraded mechanical property as shown in table 2 and 3. The yield stress and the maximal stress, deteriorate also but with a less value comparatively to the Young modulus.

#### 2.2. Ageing influence on fatigue properties

Figure 3 shows the S-N curves of the three sets of neoprene specimens used in our tests. The fatigue properties of these sets of specimens are presented in table 4.



**Figure 3** : S-N curves of the three sets of neoprene specimens To characterize the fatigue life-stress relationship of the materials, we usually use the wöhler's relation (4) :

$$LogN_{f} = a - b\sigma$$
 (4)

Where, a and b are two constants and  $\sigma$  is the amplitude of the applied stress

Table 4 : Fatigue properties of the three sets of neoprene specimens

SN curves properties Matérial	Fatigue strength $\sigma_D$ (MPa)	Failure cycle Number - Stress relationship
New specimen	1,65	$Log Nf = 22,56 - 5,4 \sigma$
Ageing specimens during 48 hours at 100 °C	1,31	Log Nf = 22,80 - 7,04 $\sigma$
Ageing specimens during 96 hours at 100 °C	1,29	Log Nf = 22,70 -7,19 σ

Figure 3 shows that the S-N curve shapes of the three sets of neoprene specimens are the same. It shows, also, that the fatigue strength brutally falls between the new specimens and the aged ones indicating a fatigue strength loss of neoprene under accelerated ageing as shown in figure 4.



Figure 4 : Fatigue strength versus ageing time

A mathematical law governing the fatigue strength versus the ageing time is proposed. It consists of experimental data collection, presented in figure 3, smoothing by a polynomial function. This relation is named characteristic function of the fatigue strength versus the ageing duration and it is written as the following expression (5):

$$\sigma(t) = 1,45 - 0.00855035 \quad t + 0,000109261 \quad t^2 \tag{5}$$

Figure 5 shows the fatigue strength of the three sets of neoprene specimens versus the ageing duration plotting with our proposed characteristic function. This figure indicates a good correlation between our experimental results and the predictive ones.

#### CONCLUSION

In this work, we show with tension tests that the neoprene degrades under ageing effect in the steam room.

This degradation appears by a decrease of its mechanical properties like Young modulus, yield stress and maximal stress of about 30%.



Figure 5 : Fatigue strength versus ageing time plotted with our proposed characteristic function

The fatigue tests confirm this assessment since the fatigue strength of the new specimens and the aged ones decrease in the same proportion. The source of these degradations is not clearly identified yet but they are probably due to chemical reactions occurring between the neoprene and its environment. These reactions may change the polymer chain length, cross-link density and/ or chemical structure.

A characteristic function of fatigue strength versus ageing time is proposed and a good correlation between our experimental results and the predictive ones obtained with this function is shown in figure 5. However, a few numbers of tests doesn't allow us to pull definitive conclusion about the reliability of this relation.

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