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Study of the fatigue behaviour of the chloroprene rubber for uniaxial tests with infrared methods

Rubber-like materials are widely used in the industry, aeronautic or automotive. In these applications, fatigue damage must be taken into account, which requires very long and costly experimental campaigns. One of the main goals of the research on this kind of materials is to improve the prediction of fatigue life with shorter and cheaper tests. This paper describes the first part of an ongoing research about the study of the fatigue behavior of dumbbell specimen made of polychloroprene with infrared thermography. It deals with the definition of two new thermal parameters for the fatigue behavior analysis. The first one is the heating rate and is defined in the first 1500 cycles giving a linear relationship with the stabilized temperature of the specimen. The second thermal parameter is the number of cycles until the stabilization of the surface temperature of the sample. Changes in the thermal behavior observed during fatigue tests,at a given load ratio F_{min}/F_{max} = 0.1, have been correlated with fatigue behavior changes.

Key words: *fatigue, infrared thermography, chloroprene rubber*

Badanie zachowania zmęczeniowego wulkanizatów kauczuku chloroprenowego metodami w podczerwieni w testach jednoosiowych naprężeń

Materiały wysokoelastyczne są szeroko stosowane w przemyśle, np. samochodowym czy lotniczym. W tych zastosowaniach musi być brane pod uwagę uszkodzenie zmęczeniowe, co wymaga zespołu bardzo długich i kosztownych badań. Jednym z głównych celów badań nad tego rodzaju materiałami jest udoskonalenie prognozowania trwałości zmęczeniowej na podstawie krótszych i tańszych testów. W artykule opisano pierwszą część prowadzonych badań, dotyczących zachowania zmęczeniowego próbki w kształcie wiosełka wykonanej z kauczuku chloroprenowego, z wykorzystaniem termografii w podczerwieni. Zdefiniowano dwa nowe parametry termiczne przydatne w analizie zachowania zmęczeniowego. Pierwszym z nich jest szybkość wydzielania się ciepła, określana w pierwszych 1500 cyklach, dająca liniowy przebieg stabilizowanej temperatury próbki. Drugim parametrem termicznym jest liczba cykli do ustabilizowania się temperatury powierzchni próbki. Zmiany w zachowaniu termicznym obserwowane podczas badań zmęczeniowych, przy danym stosunku obciążenia F_{min}/F_{max} = 0,1, zostały skorelowane ze zmianami zachowania zmęczeniowego.

Słowa kluczowe: *zmęczenie, termografia w podczerwieni, kauczuk chloroprenowy*

1. Introduction

Fatigue design is based on test campaigns to obtain representative data for material behavior which are very time and material consuming. Therefore, many studies

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have been done to find a way to get the same information faster and cheaper.

For example, the infrared thermography provided the surface temperature of the specimen and was first time used in studies about metallic materials. Luong demonstrated a link between the damage and the evolution of the temperature during a fatigue test [1] as he showed that its intrinsic dissipation was the most accurate and sensitive manifestation of damage, directly related to localized plasticity prior to crack initiation in a metallic specimen. Chrysochoos demonstrated that the heat sources were linked to the strained zones and later, how to localize precisely the stress field with an infrared camera [2 – 3]. Consequently many methodologies were developed for the fast evaluation of the fatigue behavior. The fatigue limit could be determined quite easily as shown by Krapez [4], La Rosa and Risitano [5] or Cura [6]. Eventually, it was possible to get the Wöhler curve as shown by Fargione [7], Doudard [8] or Meneghetti [9].

The same kind of studies has been carried out on polymers, but the issue with the organic materials is that on thermal measurements it is difficult to dissociate damage and other sources of dissipation (viscosity for example). Wattrisse showed that the thermal source distribution is correlated with the strain and stress fields and that for glassy polymers there is a predominance of thermoelastic effects over viscous effects in the heating process [10]. Jegou then demonstrated that by using only infrared thermography it was possible to predict a Wöhler curve for short fiber reinforced plastic [11].

The Infrared thermography has been less used on elastomers because it is more difficult to link the evolution of the self-heating and the damaging processes.

During a fatigue test, the heat generation is the conversion of the mechanical energy into heat due to the hysteretic behavior. Many works have been done on the causes and consequences of heat generation of natural rubber. The reticulation has been shown to decrease the self-heating [12] as it limits the displacement, and therefore the friction, between the chains. In the same paper, the author described the impact of the heat on the mechanical characteristics: the higher is the temperature of the specimen, the lower are the failure and abrasive properties, but also the lower are the static and dynamic moduli and the tangent delta. For carbon black particles, it was found that at low deformation they have almost no impact on the self-heating. However for higher deformation, the difference of modulus between the filler and the rubber matrix becoming very important, there are locally very high strains around the particles which lead to an increase of the hysteresis, hence of the self-heating [13]. Kar could relate numerous parameters to the variation of the self-heating as for example the increase of the hysteresis or the frequency leads to an increase of the self-heating [14]. Harbour [15] showed that the evolution of the stabilized surface temperature with the hysteresis area follows a linear relationship for uniaxial and multiaxial loadings and whether the rubber used crystallizes or not during fatigue tests.

Heat generation not being an intrinsic characteristic, as it is very shape-dependant, other parameters linked to the self-heating were investigated. That is how Le Saux designed a fatigue criterion coupling infrared measurements with X-ray tomography on polychloroprene rubber [16].

The results about the study of the fatigue of some material with infrared thermography show the necessity to wait until the stabilization of the specimen temperature. For thin geometries or high thermal diffusivity material it is not a real problem because the stabilization is quite fast. On the contrary, for rubber specimen with a more important volume, the time required for having the surface temperature stabilized is much more than for thinner metallic materials.

This paper is about a faster way to get the informations on the thermal behaviour during a fatigue test for a uniaxial test at a given load ratio and some pertinent results about the fatigue behavior of the polychloroprene rubber.

In the first part, the experimental setup of the fatigue campaign and the material studied are described. The parameters investigated in this paper are defined in the second part. Then, in the third part, the results of the campaign at a 0.1 load ratio are presented and analyzed.

2. Experimental setup

This paper investigates the fatigue behavior of a polychloroprene rubber with infrared thermography. Therefore, every single fatigue test carried out in this study has both mechanical and thermal angles.

2.1. Polychloroprene rubber

The material studied in this paper is a vulcanized polychloroprene rubber (CR) filled with N990 carbon black (table 1). The specimen used is of a dumbbell shape made of a rubber part 30 mm long, bounded to two metal parts at each extremity which can be attached to the fatigue machine with screws (Fig 1). Those specimens were molded by an injection press device at 175°C, during 4 minutes.

Table 1. *Short details about the formulation of the CR*
Tabela 1. *Niektóre dane dotyczące składu mieszanek CR*

Elastomer	CR type G
Filler	Thermal Carbon Black (N990)
Curative system	S-ZnO-MgO

2.2. Mechanical tests

The fatigue test campaign was carried on an INSTRON 8802 servo-hydraulic fatigue testing machineat

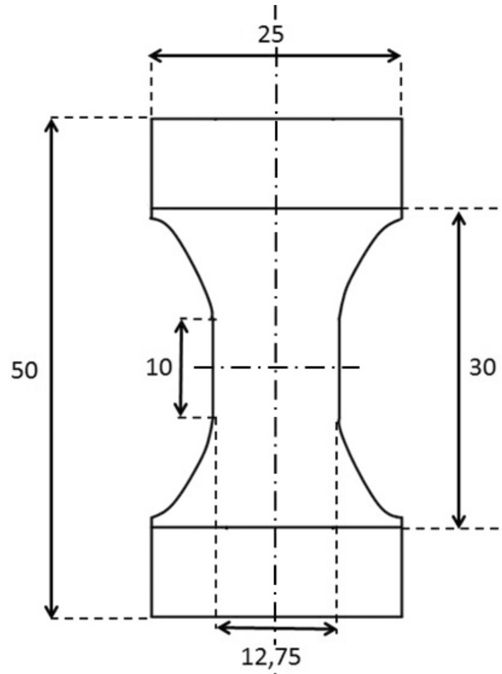

Figure 1. *Dumbbell-type specimen – length in mm*
Rys. 1. *Próbka typu wiosełko – wymiary w mm*

room temperature. It consisted of load controlled fatigue tests at 5 Hz and at a load ratio R of 0.1 with F_{max} the maximum load during the test and F_{min} the minimum load:

$$R = \frac{F_{min}}{F_{max}} \qquad (1)$$

The amplitude stress σ_a is defined by the equation (2), with S the area of the cross section of the median part of the specimen. The range of amplitude stresses which have been investigated are given in the table 2. The fatigue data gathered by Poisson [17] or Lacroix [18] show that, depending on the loading conditions, tests can fail after a few thousand cycles or more than one million cycles, depending upon the stress amplitude.

$$\sigma a = \frac{F_{max}}{2S(1-R)} \qquad (2)$$

Table 2. *Range of amplitude stresses tested*
Tabela 2. *Zakres amplitudy badanych naprężeń*

F_{max} (N)	85	100	115	125	140	150	160	175
σ_a (MPa)	0.32	0.38	0.44	0.48	0.53	0.57	0.61	0.67

Tests have been carried out until either failure or stabilization of the surface temperature (if the fatigue life of the specimen was longer than 10^5 cycles). The previous works on the fatigue behavior of a polychloroprene dumbbell specimen provide a fatigue life data base which will be compared to the present results.

Three tests were carried out for each mechanical condition to investigate the dispersion of the fatigue results.

2.3. Thermal measurements

The thermal measurements have been conducted with an infrared camera CEDIP Jade III MWIR (InSb) operating under an acquisition frequency of 50 Hz. The focal plane array is a 320×240 array of detectors digitized on 14 bits and sensitive in the 3.6 – 5.1 μm spectral band wave-lengths. Before any measurement, a calibration was realized to check the conversion of the signal into temperature (in °C). To do so, a black body was used and a 2 points non uniformity correction (NUC) was applied to the array of detectors. After the calibration, a 25 mK precision is obtained. To minimize the influence of the external environment on the measurement, a black sheet was placed around the fatigue machine.

In order to have a precise idea of the evolution of the temperature during a fatigue test and because of the limited memory capacity of the camera, it was decided to use the sampling function of the camera: instead of recording 50 images per second (ips), it would record 1 or 2 ips. The variation of the temperaturefor a cycle is very low given the mean temperature which is the parameter investigated. The loss of precision caused by the sampling procedure is therefore limited.

The camera records an image with a 320×240 pixels size in which the surface temperature field of the specimen can be seen (fig. 2). With this figure, it is possible to measure several values which will be useful for the analysis of the campaign. Three boxes are used to measure

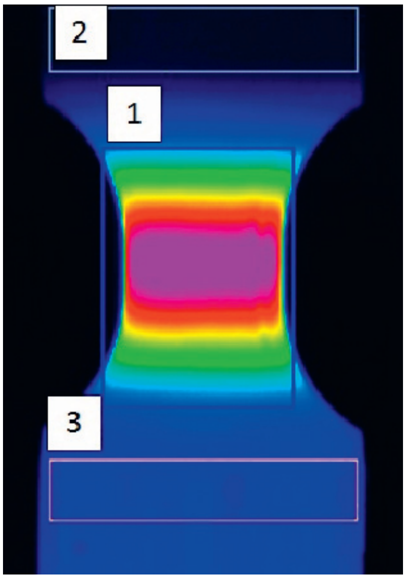

Figure 2. *Image of the specimen during a test given by the infrared camera with the localization of the boxes used to measure the thermal parameters: T_{max} measured in the box 1, T_{top} in the box 2 and T_{bot} in the box 3.*
Rys. 2. *Obraz próbki podczas rejestrowania badania przez kamerę na podczerwień z lokalizacją komór używanych do mierzenia parametrów termicznych: T_{max} mierzona w komorze 1,T_{top} – w komorze 2 i T_{bot} – w komorze 3*

specific temperatures: the box 1 measures the maximal surface temperature of the specimen T_{max}; the box 2 measures the temperature of the top clamp T_{top} and box 3 the bottom clamp temperature T_{bot}.

With these temperatures, it is possible to define the thermal parameters used to analyze the data from the fatigue campaign.

3. Definitions

The thermal parameters investigated in this paper are defined hereafter (figure 3) thanks to the measurement of the maximum surface temperature and the temperature of the two clamps.

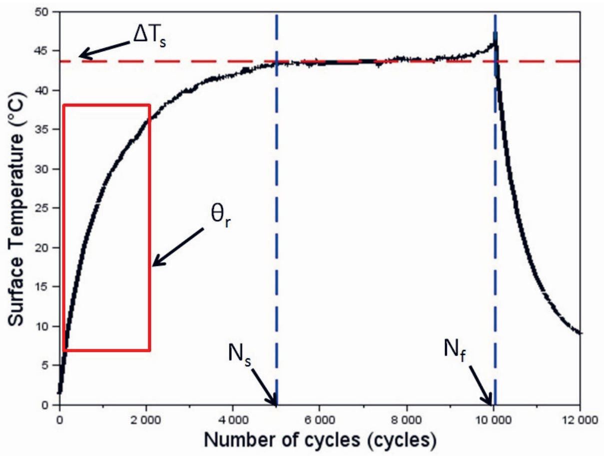

Figure 3. *Evolution of the surface temperature during a fatigue test*
Rys. 3. *Zmiana temperatury powierzchni podczas próby zmęczeniowej*

The surface temperature at the N^{th} cycle ΔT (N) is defined by the following relation (3). It takes into account any variation of the ambient temperature or any heat flux from the specimen to the clamps.

$$\Delta T(N) = T_{\max(N)} - \frac{T_{top} + T_{bot}}{2} \quad (3)$$

N_s represent the number of cycles needed to obtain the stabilization of the surface temperature on the specimen.

The fatigue life of the specimen N_f is the number of cycles until failure of the specimen.

The stabilized temperature ΔT_s is defined as the mean value of the surface temperature taken between N_s and N_f (4).

$$\Delta T_s = average(\Delta T(N);\ N \in [N_s; N_f]) \quad (4)$$

Finally, inspired by what Zhang [19] did on metallic materials, the heating rate θ_r has been defined as the slope of the temperature during a fatigue test between the 100^{th} and 1500^{th} cycles in a logarithmic base (fig. 4). It is expressed in °C/decade.

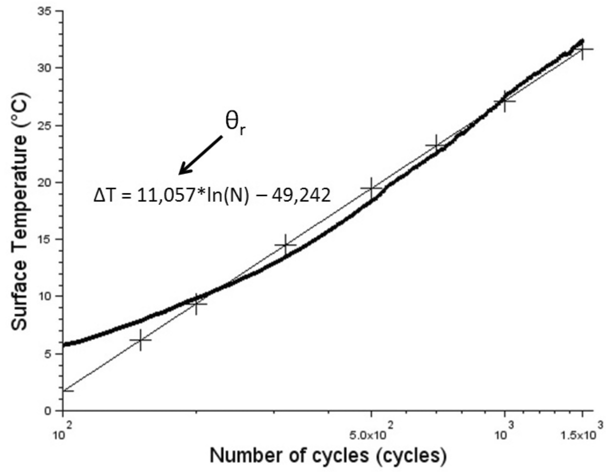

Figure 4. *Evolution of the surface temperature between 100 and 1500 cycles in a log base*
Rys. 4. *Zmiana temperatury powierzchni w zakresie 100 – 1500 cykli zmęczeniowych, w skali logarytmicznej*

The thermal parameters thus defined, the results from the uniaxial fatigue campaign can now be analyzed.

4. Results

Monitoring the evolution of the temperature during a fatigue test gives access to the parameters defined in the previous paragraph and, then, several relationships can be highlighted.

4.1. Relationship between heating rate and stabilized temperature

On the figure 5, the evolution of the surface temperature at the beginning of the fatigue test for several stress amplitudes is depicted. The first result that can be deduced is that the higher the stress amplitude, the higher the stabilized temperature and the heating rate.

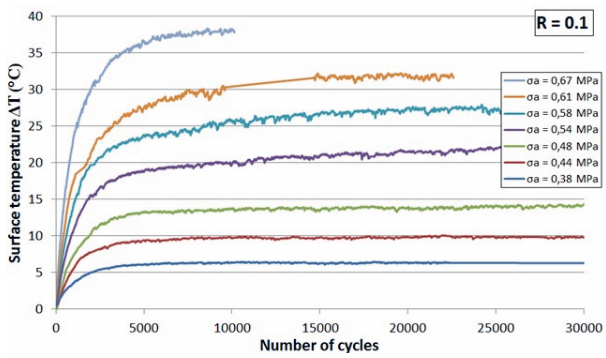

Figure 5. *Evolution of the surface temperature for several stress amplitudes*
Rys. 5. *Zmiana temperatury powierzchni dla poszczególnych amplitud naprężenia*

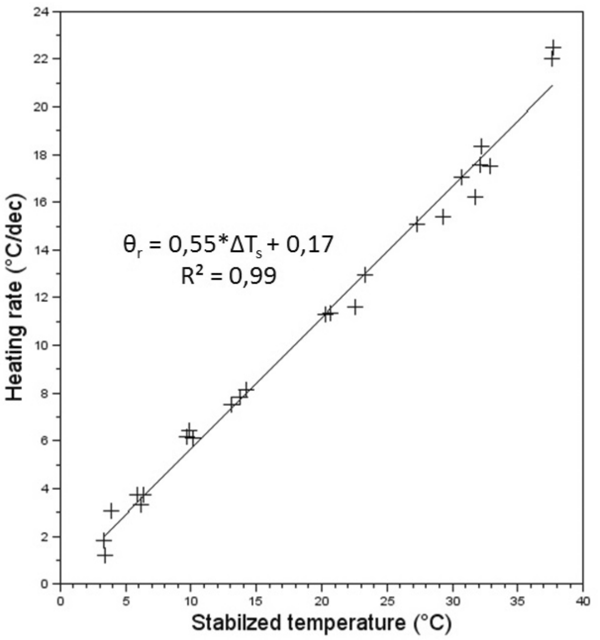

Figure 6. *Evolution of the heating rate with the stabilized temperature*

Rys. 6. *Zmiana wydajności generowania ciepła wraz ze stabilizowaną temperaturą*

However the stabilization of the surface temperature can take a long time for lower stress amplitudes (up to 10^5 cycles). A solution to this problem is given in figure 6, showing the evolution of the heating rate with the stabilized temperature. It can be seen that there is a linear relation between the two parameters with a very good precision (R^2 = 0.99). The heating rate shows a lower dispersion than the stabilized temperature and is also quicker and easier to measure.

This result is quite interesting because it means that, with such a specimen, in the first 1500 cycles there is an access to the information given by the stabilized temperature many cycles later.

Given this relationship, the next figures involving the temperature will be drawn using the heating rate.

4.2. Fatigue behaviour

N_s versus N_f have been plot on the figure 7 for each tests which were carried out until failure. There seems to be a power law relationship with a good accuracy given the dispersive nature of the two parameters for a load ratio of 0.1.

It raises a question: as the Wöhler curves represent stress versus the number of cycles until failure N_f, what could be the result if a thermal parameter was drawn versus the number of cycles until the surface temperature stabilization N_s?

To exploit this aspect, Figure 8 shows the evolution of the heating rate versus the number of cycles until the surface temperature stabilization N_s. There is a clear change in the thermal behavior around a stress amplitude of 0.44 MPa: indeed for higher amplitude stress, the points are on the same correlation line and for the lower amplitude stress the number of cycles until the surface temperature stabilization is becoming higher and higher. It is worth noting that for the σ_a < 0.44 MPa, no failure is observed before 10^6 cycles.

The next step is to compare this result with a common Wöhler curve determined by Lacroix [18] (fig. 9). As one can see, there is no failure until 10^6 cycles for amplitude stress lower than 0.46 MPa. This is close to the value for which a thermal behavior change has been

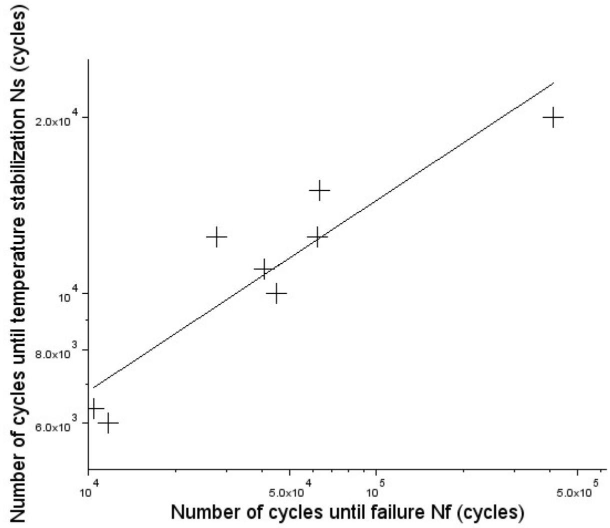

Figure 7. *Evolution of the number of cycles to stabilize the temperature of the specimen N_s with the fatigue life N_r*

Rys. 7. *Zmiana liczby cykli do ustabilizowania temperatury próbki N_s w stosunku do trwałości zmęczeniowej N_f*

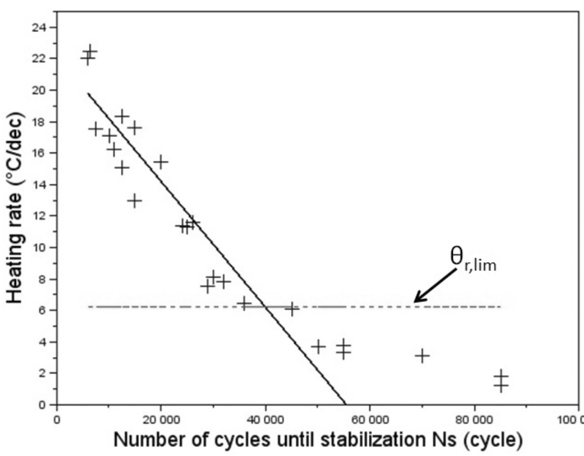

Figure 8. *Evolution of the heating rate with the number of cycles until temperature stabilization at a load ratio 0.1*

Rys. 8. *Zmiana wydajności generowania ciepła (°C/dekadę) w stosunku do liczby cykli do ustabilizowania temperatury przy stosunku obciążenia 0,1*

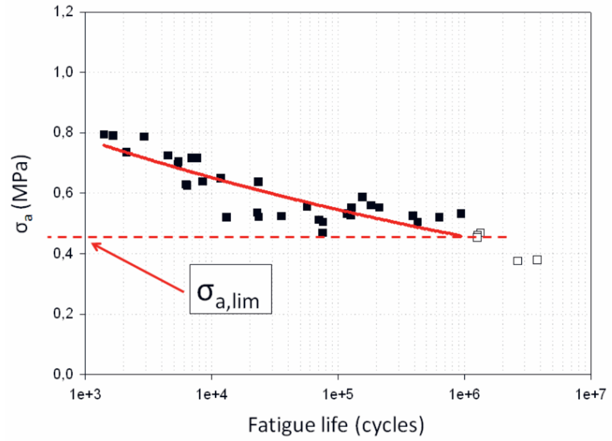

Figure 9. *Wöhler curve of the CR specimen plotting the alternate stress versus the fatigue life [20]*
Rys. 9. *Krzywa Wöhlera próbki CR wykreślona dla kolejnych naprężeń w funkcji trwałości zmęczeniowej [20]*

observed in our tests (fig. 8). Therefore, there seems to be a correlation between a thermal behaviour change and a fatigue behaviour change.

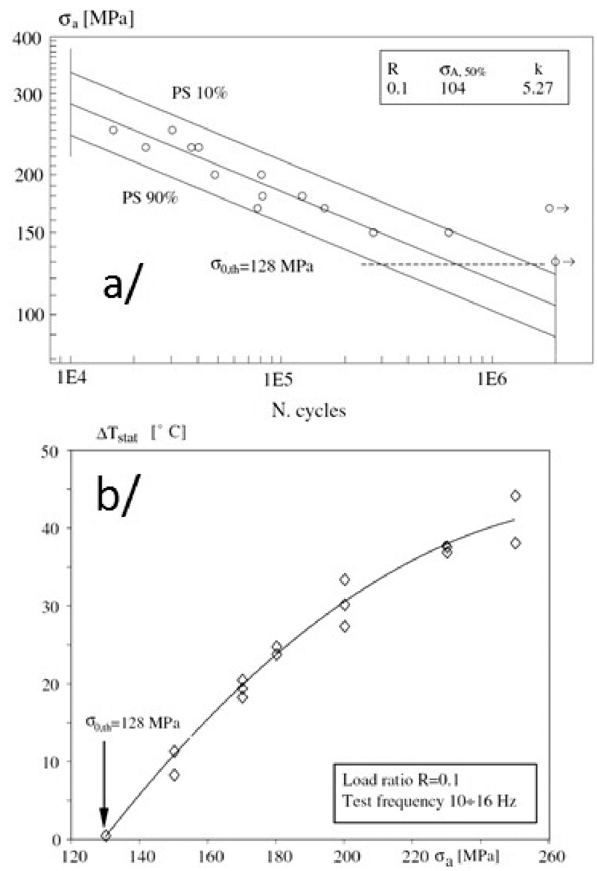

Figure 10. *Determination of the endurance limit by Meneghetti [7] comparing of Wöhler curve (a) and infrared measurements (b)*
Rys. 10. *Porównanie granicy wytrzymałości ustalonej przez Meneghettiego [7]: wykres Wöhlera (a) i pomiary w podczerwieni (b)*

This result can be found with another approach. For the metallic materials, Meneghetti [9] showed that it was possible to measure the endurance limit through infrared measurements (fig. 10). He first drew a Wöhler curve to get the endurance limit (fig. 10.a). Then on a figure showing the evolution of the stabilized temperature versus the amplitude stress (fig. 10.b), he found that the intersection of the correlation line of thermal measurements and the abscissa has the same value as the endurance limit.

Then, drawing the evolution of the heating rate versus the amplitude stress (fig. 11) and the correlation line for the more damaging tests shows that this method does not suit the elastomer [20]. Indeed with the thermal approach, if we applied the same protocol used in [8] or [9], it appears that the value picked from the intersection between the correlation line and the abscissa (σ_a = 0.36 MPa – fig. 11) is different with the one get from fatigue data (σ_a = 0.44 MPa – fig. 12).

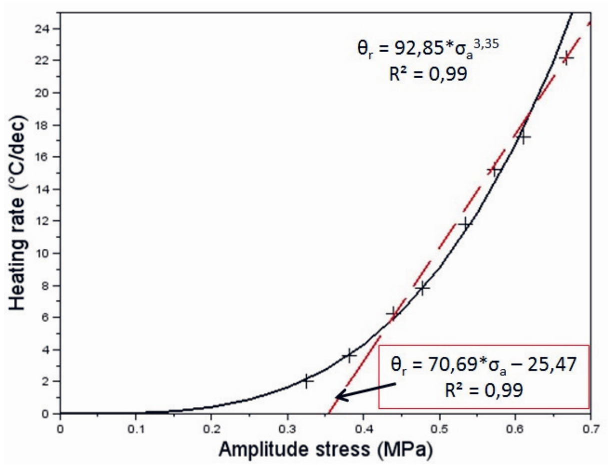

Figure 11. *Evolution of the heating rate versus the amplitude stress*
Rys. 11. *Zmiana wydajności generowania ciepła w funkcji amplitudy naprężenia*

Thus, there are two fatigue behaviours which are observed, depending whether a test is at a σ_a lower or higher than 0.44 MPa. There is no failure before 10^6 cycles for amplitude stress lower than 0.44 MPa and given the nonlinear evolution of the curve at lower stress amplitudes, it appears that the heat build-up seems to be mainly caused by the viscous behavior, at low stress amplitudes. However, for higher stress amplitudes, the numerous failures before 10^6 cycles and the linear evolution of the curve could be correlated mainly to the damaging process than the effect of viscosity at lower stresses.

A new definition of the fatigue limituntil 10^6 cycles can be proposed as the alternate stress at which the fatigue behaviour of the specimen changes is measured, in this case 0.44 MPa, which correlates well with our the fatigue data base [18].

Moreover, the use of the heating rate can be interesting when it comes to pre sizing given that it allows getting access to mechanical data faster than a classical fatigue campaign.

4.3. Hysteresis area

The hysteresis area can be used in modeling the fatigue life of elastomer materials as shown by Poisson [20]. It is then interesting to see that it can be measured thanks to thermal measurements.

It is not new; we can quote Harbour [15] who showed that there is a relationship between the stabilized temperature and the dissipation rate (defined as the hysteresis area time the frequency of the test) for SBR and NR and several types of mechanical loadings. Le Saux [20] found a similar relationship for the CR.

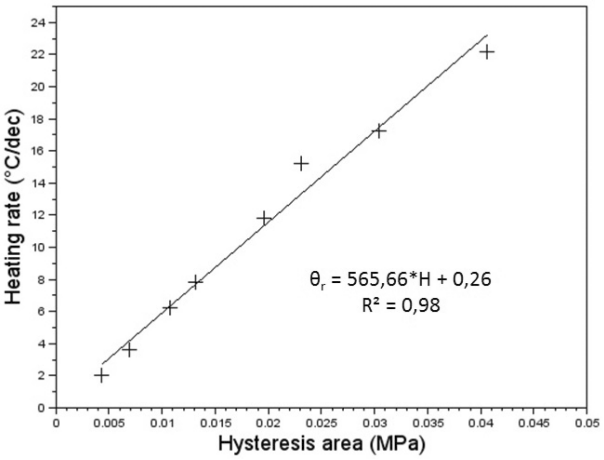

Figure 12. *Evolution of the heating rate versus the hysteresis area*
Rys. 12. *Zmiana wydajności generowania ciepła w funkcji wielkości pola histerezy*

However, it was necessary to wait until the stabilization of the surface temperature. In our case, the figure 12 plotting the evolution of the heating rate versus the hysteresis area shows the same linear relationship.

5. Conclusion

New fatigue parameters have been introduced in this paper: the heating rate and the number of cycles until the stabilization of the surface temperature on a chloroprene rubber.

The heating rate is very easy and fast to measure and its relationship with the stabilized temperature could be interesting due to the time saving possibilities it implies.

The number of cycles until the stabilization of the surface temperature has shown some interesting link with the number of cycles until failure.

It was then possible to see a correlation between a thermal behaviour change and a fatigue behaviour change.

We propose a new definition for the fatigue limit until 10^6 cycles, being the alternate stress around which the thermal behaviour of the material changes.

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References

1. *Luong M.P., Mech. of Mat. 1998, 28, 155.*
2. *Chrysochoos A., Louche H., C.R. Acad. Sci. Paris, 1998, 326, 345.*
3. *Chrysoochos A., Louche H., Int. Jour. of Eng. Sc., 2000, 38, 1759.*
4. *Krapez J.-C., Pacou D., Gardette G., Proceeding of Quantitative Infrared Thermography held in Reims, 2000, 277.*
5. *La Rosa G., Risitano A., Int. Jour. of Fat. 2000, 22, 65.*
6. *Cura F., Curti G., Sesana R., Int. Jour. of Fat. 2005, 27, 453.*
7. *Fargione G., Geraci A., La Rosa G., Risitano A., Int. Jour. of Fat. 2002, 24, 11.*
8. *Doudard C., Calloch S., Cugy P., Galtier A., Hild F., Fat.&Fract. of Eng. Mat.&Struct. 2005, 28, 279.*
9. *Meneghetti G., Int. Jour. of Fat. 2007, 29, 81.*
10. *Wattrisse B., Muracciole J.-M., Chrysochoos A., Int. Jour. of Ther. Sc. 2002, 41, 422.*
11. *Jegou L., Marco Y., Le Saux V., Calloch S., Int. Jour. of Fat. 2012, 47, 259.*
12. *Medalia A.I., Rubb. Chem. and Tech. 1991, 64, 481.*
13. *Meinecke E., Rubb. Chem. and Tech. 1991, 64, 269.*
14. *Kar K.K., Bhomwick A.K., Jour. of App. Pol. Sc. 1997, 64, 1541.*
15. *Harbour R.J., Fatemi, A., Mars, W.V., Int. Jour. of Fat., 2008, 30, 1231.*
16. *Le Saux, V., Marco Y., Calloch S., Doudar C., Charrier P., Proc. Eng., 2010, 2, 949.*
17. *Poisson J.L., Lacroix F., Meo S., Berton G., Ranganthan N., Int. Jour. of Fat. 2011, 33, 1151.*
18. *Lacroix F., «Etude du comportement en fatigue des caoutchous synthétiques», Université François Rabelais, Tours 2004, 208.*
19. *Zhang L., Liu X.S., Wu S.H., Ma Z.Q., Fang H.Y., Int. Jour. of Fat., 2013, 54, 1.*
20. *Le Saux V., «Fatigue et vieillissement des élastomeres en environnement marin et thermique: de la caractérisation au calcul de structure», Université de Bretagne Occidentale, Brest 2010, 290.*