

A NEW APPROACH FOR CRACK GROWTH LIFE OF AN ELASTOMERIC MATERIAL

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Abstract

Manufacturing process of elastomers is a fundamental aspect for the rubber industry. Manufacturing elastomers involves defect in the rubber blends. These defects should have an impact the fatigue life of rubber. So, the determination of a defect size threshold – below which the defect size has no impact on fatigue life – becomes a crucial point. However, the application of fracture mechanics to the study of rubber involves some difficulties because of the large deformations occurring in the rubber. Moreover, as fatigue damage of elastomers depends not only upon the mechanical loading (stress level, frequency and loading ratio), but also on thermal damage (temperature and thermal dissipation) and chemical damage (oxygen and ozone), the study of viscoelastic behaviour seem to be a promising way to understand fatigue behaviour of elastomers. In the present work crack propagation tests using a DMA (Dynamic Mechanical Analyser) have been carried out and evolution of the viscoelastic properties has been measured. At the same time a rheological study has been carried out to look at the influence of temperature, frequency, and stress level on crack growth propagation.

1. INTRODUCTION

Many product failures arise from defects, so understanding crack propagation is very important in using rubbers in applications involving fatigue. As in the case of metals, studies of fatigue have to be separated in two parts: nucleation of cracks and then their propagation. The application of fracture mechanics to rubber involves some difficulties because of the large deformations occurring in rubber. Indeed, the elasticity of elastomers is not linear and moreover, highly deformed cracks do not stay sharp as in the Griffith's model (*Bathias et al.* [1]). Fatigue damage of elastomers depends not only upon the mechanical loading (stress level, frequency and loading ratio), but also on thermal damage (temperature and thermal dissipation) and chemical damage (oxygen and ozone) (*Legorju and Bathias* [2]). Filled vulcanized elastomers are known to exhibit a complex rheological behavior. This behavior is characterized, in particular, by a high level of non linear viscoelasticity. Thermal dissipation which occurs for dynamic loading is an important aspect. Indeed, the increase of the temperature leads to a modification of the properties and can damage the material. The viscoelasticity of elastomers is a parameter which could influence the fatigue life. Crack growth propagation tests have been carried out on a strain-controlled DMA (Dynamic Mechanical Analyser) specifically designed for polymers (TA Instrument 2980).

Viscoelastic properties of different types of elastomers, with fatigue loading, were already studied using DMA in our previous work. The results (*Lacroix et al. [3]*) indicate a quick way of identifying fatigue resistant elastomers. In addition to certain studies on crack nucleation of CR (*Lacroix et al. [4]*) showing the drastic influence of the defect size on fatigue life (Post Mortem analysis on fracture rupture of dumbbell specimen had been performed by Scanning Electron Microscopy analysis (SEM)), crack propagation studies have also been investigated to evaluate the effect of the defect size on the fatigue life. This approach is similar to that of Murakami (*Murakami et al. [5]*) for metals. Tests were conducted on small specimens with different initial flaw sizes, created by a razor blade. These first results confirm that the defect size has an influence on fatigue life and show that below a particular flaw size, fatigue life drastically increases, for a given stress level. The present work also shows the possibilities of quantitative fatigue studies using a DMA (at a maximum load of 18 Newtons) to understand the fatigue resistance of elastomers.

2. EXPERIMENTAL

Materials

All tests were performed with a series of chloroprene rubber (CR) and hydrogenated nitrile butadiene rubber (HNBR). The static mechanical properties for both rubbers are shown in Table 1. For the characterisation, we used specimens having the same dimensions as for the crack propagation tests, but without cracks. For the crack propagation study, we used an Edge Cracked Simple Tension specimen (E.C.S.T) (thickness = 2 mm, width = 9 mm) with a crack initiated on one edge of the sample. DMA film specimens (ECST) were prepared using a cutter constructed with two sharp parallel blades. The study of the influence of crack size on fatigue has been carried out making different crack lengths of 0.35 mm to 2 mm achieved with a special device using a razor blade. The accuracy of the mechanical crack length is in the range of ± 0.1 mm.

TABLE 1. Material properties.

Material	Young's Modulus / MPa	Rupture elongation / (%)	Hardness / Shore A
CR	3,8	680	54
HNBR	3,9	407	50

Methods

The behaviour of a linear viscoelastic material is characterised by properties such as elastic and loss moduli. These properties represent the ability to store and dissipate energy in dynamic testing and are obtained by measuring the force and the displacement developed by the material when subjected to an oscillatory deformation of controlled amplitude and frequency.

A TA Instrument DMA 2980, specially designed for such tests, was employed to investigate the viscoelastic properties of CR and HNBR mentioned previously. The instrument calibration of some parameters was performed and this included the DMA firmware, height, force, instrument rigidity and self deformation measurement, clamp weight compensation, temperature, and furnace. Tests were performed in displacement control. The temperature

was maintained by enclosing the sample in a small forced gas convection oven. Oven stability was to $\pm 1^\circ\text{C}$ of the temperature setting, and equilibrium was rapid. The specimen was mounted between special tension clamps and was carefully aligned to avoid any twisting or buckling of the sample whilst under any force. Once mounted, the actual length between clamps is automatically recorded. For characterisation of viscoelastic properties some additional controls were used on DMA 2980 TA system with options available for static and dynamic control of the sample. The static force is required to maintain contact between the sample and the measuring fixture and should not be excessive in order to avoid sample deformation, bending or buckling under higher force. A fixed force can be used throughout the entire scans although other controls that include tension and position control are also available. Under tension control, the static force is applied to the sample maintaining a constant relationship between the static and dynamic forces during the scans. Moreover, the position control mode has the static force applied to the sample in order to maintain the sample at a specific set point or position. For our study, the dynamic displacement amplitude was selected and the strain control maintains the strain constant by adjusting the dynamic force. Moreover, the relationship between the static and dynamic forces was selected such that the static force was 1.25 times the dynamic force during the scan.

3. RESULTS AND DISCUSSION

Viscoelastic behaviour

Before performing crack growth tests, it was necessary to look at the dynamic properties of both rubbers. First it is necessary to ensure that the properties were in the same range of values with respect to the frequency, temperature and dynamic amplitude, then to link fatigue and viscoelastic behaviour, and finally to determine a viscoelastic model (this part is not treated in this paper). The characterisation of viscoelastic properties can be investigated on DMA by performing different dynamic tests on sample without crack. A first approach is to determine the linear and non linear viscoelastic behaviour by performing oscillatory tests at a given frequency and room temperature, with different strain amplitudes, ε_o , imposed. The evolution of the measured elastic modulus $E'(\varepsilon_o)$ with the amplitude ε_o at a frequency $f = 30$ Hz is shown in Fig.1-a. for the rubbers. In all the cases, a linear viscoelastic behaviour corresponding to an elastic modulus independent of the strain ($E' = E'(t)$) can be determined for small deformations. The critical deformation above which the elastic modulus decreases with increasing ε_o is determined by the onset of the non linear viscoelastic behaviour. Both the samples show a similar behaviour (Fig 1-a) in both linear and non-linear area and the range of the linear viscoelastic behaviour is roughly the same ($\varepsilon_o = 1\%$). Fatigue crack propagation tests were carried out for a dynamic strain of 10 % (because this is the typical strain range in applications), so a non linear viscoelastic model is needed. Moreover the complex modulus for these tests is comparable to the static modulus. The loss modulus of the HNBR is higher than the CR. HNBR rubber shows a stronger propensity to warm up than the CR. Damage by internal heating will be more important for the HNBR. Globally, the dynamic behavior is similar for both elastomers.

The frequency sweep shows a typical behaviour (Fig 2-a) for elastomers where the stiffness of both the rubbers increases when the frequency increases. The increase is more significant for the HNBR than for the CR. For $f = 30$ Hz – conditions of our crack propagation tests – the elastic modulus is higher for the HNBR, so for strain-controlled tests the stress will be more

significant resulting in more fatigue damage. The elastic modulus evolution versus temperature shows a similar evolution (Fig 2-b) for both rubbers particularly at 80°C (which correspond to the working temperature for these materials in structural application).

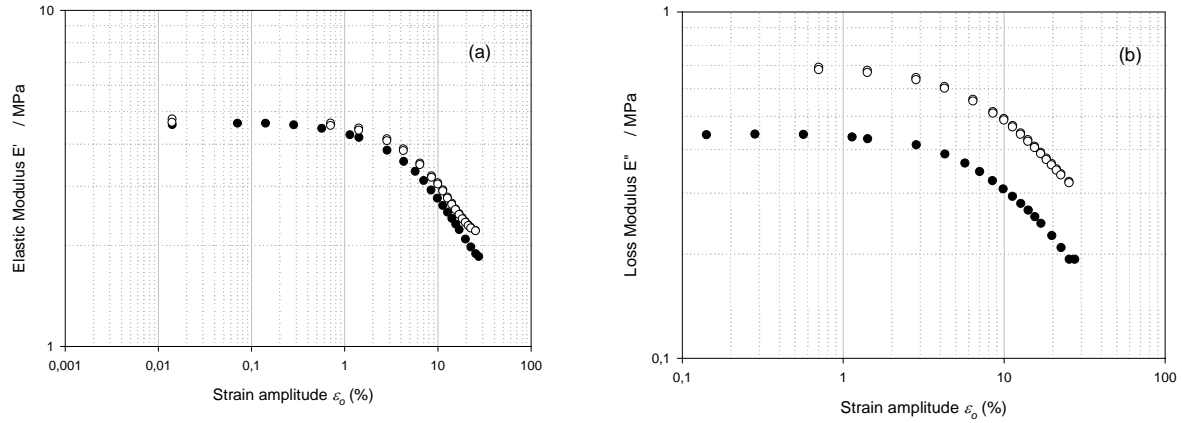


FIGURE 1. Evolution of the elastic (a) and loss modulus (b) with the strain amplitude ($f = 30$ Hz) for: (●) chloroprene rubber, (○) hydrogenated nitrile butadiene rubber.

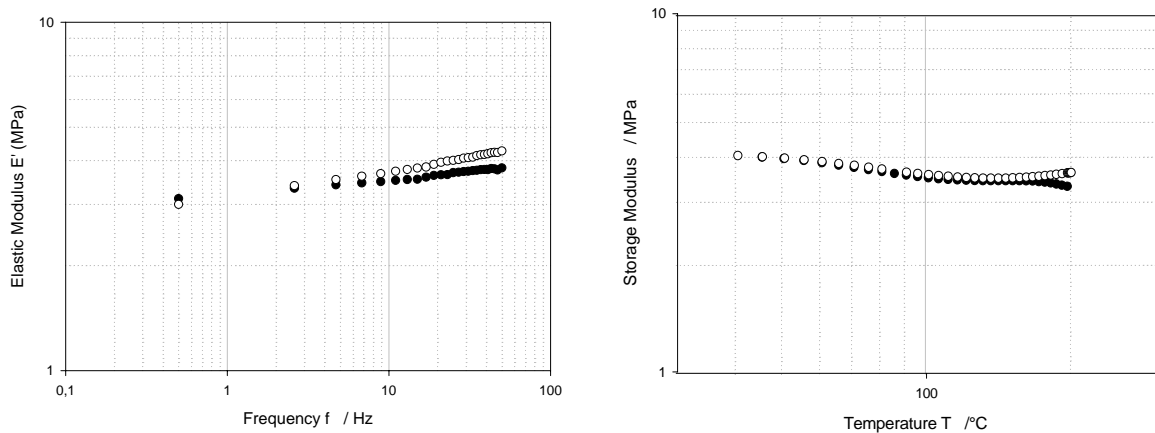


FIGURE 2. Evolution of the elastic modulus with frequency (a) ($\varepsilon_0 = 5\%$ -40°C) and with the temperature (b) ($\varepsilon_0 = 5\%$ - $f = 30$ Hz) for: (●) chloroprene rubber, (○) hydrogenated nitrile butadiene rubber.

Dependence on crack size

The influence of the defect size on fatigue has been reported in our earlier work [4] for fatigue crack nucleation tests on dumbbell (CR) specimens. Examination of the SEM micrographs of the fractured surfaces clearly indicated that the defect size had an influence on the fatigue life of the rubber. However, the problem was the impossibility to control the size of the flaw (post mortem analysis). So, a surface threshold - below which the defect size does not have an impact on the fatigue life - could not be determined as Murakami did [5].

In order to study the influence of the defect size on fatigue life, present study has been carried out on small coupons (ECST) with different initial flaw sizes, created by a razor blade. For

each test, the decrease of the elastic modulus had been recorded (Fig 3.). The number of cycles shown in Fig 4., refer to the number when the crack reached the middle of the specimen. When the crack reached the middle of the sample the stiffness is decreased by a ratio of 30% in comparison with the initial modulus. These first results show that the defect size has an influence on fatigue life and also below a particular size fatigue life drastically increases, for a given stress level. Experiments which had been carried out for a temperature of 80°C are reported in this paper. The results of the tests (Fig 4.) show a decrease of the number of cycles with the increase of the defect size for both rubbers. There is a change of slope for both rubbers for a defect size in the range of 0.5-1 mm² indicating that the proposition of the existence of a surface threshold is possible. Although, mechanical techniques for generating initial crack are not enough accurate to make smaller cracks which would allow determination of a precise surface threshold for rubber. Determination of this threshold – below which the size of defect has no influence on fatigue life - is an important point for rubber industries to improve the process fabrication of elastomers blends. After all, we can note (Fig 3. - Fig 6.) that the crack velocity of the CR is lower than the HNBR. Indeed, the fatigue life of CR is higher than the HNBR (Fig 3.). The difference between both rubbers could be explained by the ability for HNBR to generate more viscous dissipation (Fig 1(b)) for dynamics tests and therefore could lead to higher damage

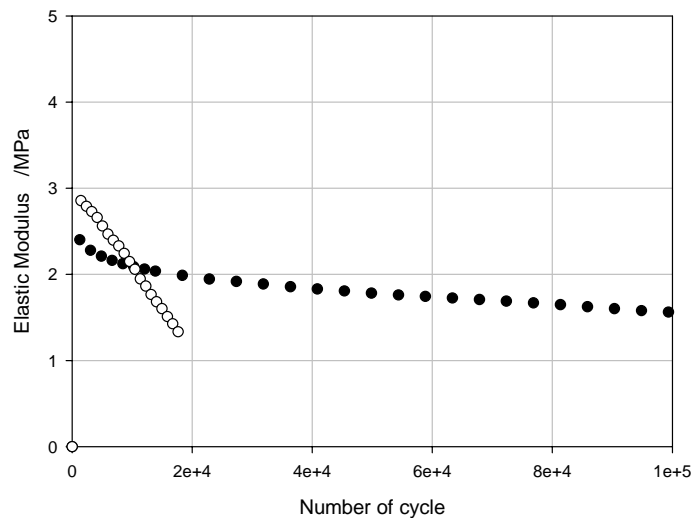


FIGURE 3. Evolution of the elastic modulus with respect to the number of cycles ($\epsilon_0 = 10\%$ - $f = 30$ Hz - 80°C) for: (●) chloroprene rubber, (○) hydrogenated nitrile butadiene rubber recorded on DMA TA for an initial crack of 1.27 mm

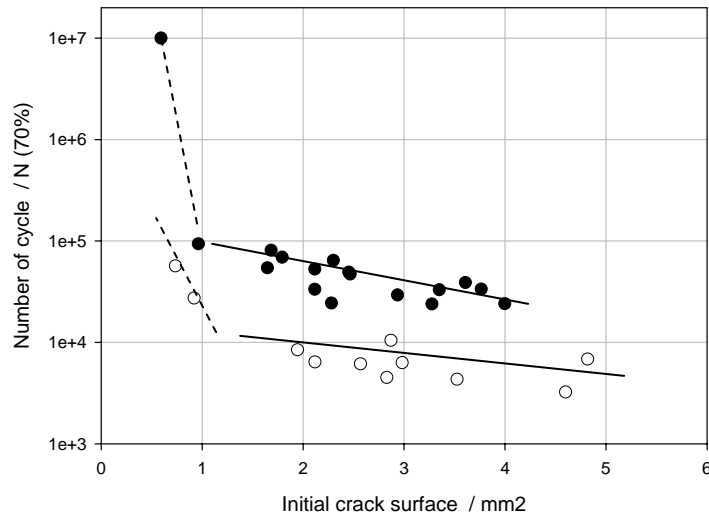


FIGURE 4. Number of cycle at 70% of stiffness versus initial crack surface ($\epsilon_0=10\%$ - $f=30\text{Hz}$ – 80°C) for: (●) chloroprene rubber, (○) hydrogenated nitrile butadiene rubber.

Two micrographs are represented in Fig. 5 showing some typical fracture surfaces of DMA specimens which represent the stable propagation of crack. Roughness is one of the main characteristics of stable propagation. Roughness is induced by secondary cracks, which are initiated near the stress concentration areas and which join the main crack, inducing fracture along several planes. For each crack propagation tests (for different crack size), we recorded for both rubbers the slope of the curve showing the evolution of elastic modulus versus the time (Fig 4.). It appears that crack velocity (Fig 7.) of both rubbers (the crack propagation velocity of HNBR is ten times higher than for CR) is linked with the roughness. Indeed, SEM observations illustrate that the roughness of the CR (Fig 5-a.) is more significant than the HNBR (Fig 5-b.), indicating a higher crack propagation velocity for the HNBR. It is in accordance with the crack propagation data (Fig 7.).

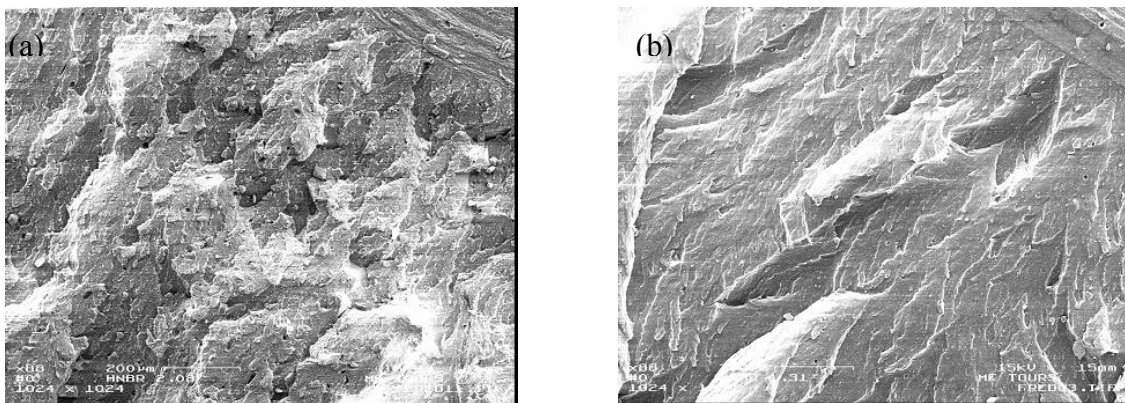


FIGURE 5. SEM micrographs of fracture surfaces of rubbers specimens: chloroprene rubber (a) and hydrogenated nitrile butadiene rubber (b).

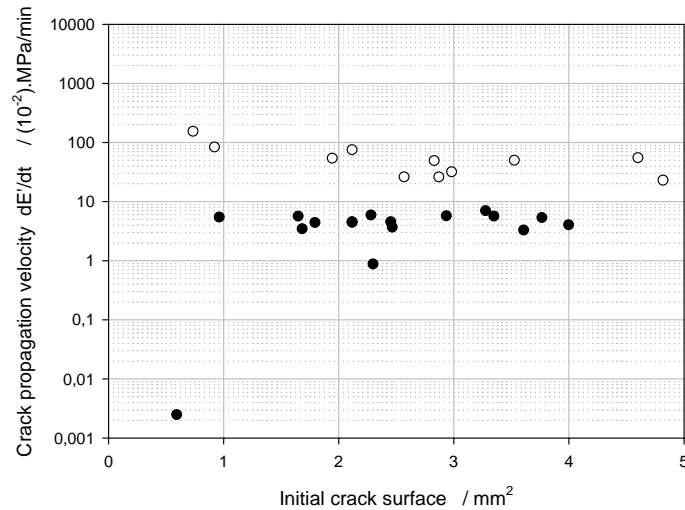


FIGURE 6. Crack propagation velocity versus initial crack surface ($\epsilon_0=10\%$ - $f=30\text{Hz}$ – 80°C) for the two elastomers: (●) chloroprene rubber, (○) hydrogenated nitrile butadiene rubber.

Dependence on temperature and frequency

Influence of temperature and frequency on crack propagation has also been investigated in the present study on the DMA 2980. Crack lengths of 1 mm were made using a razor blade. In order to study the influence of temperature on crack growth, tests were carried out at a frequency of 30 Hz and a dynamic strain $\epsilon = 10\%$. The results are shown in Figure 7 on a semi-log scale by plotting the number of cycles to failure versus the temperature. It is evident that the fatigue life of samples increases when temperature decreases in the same way for both rubbers.

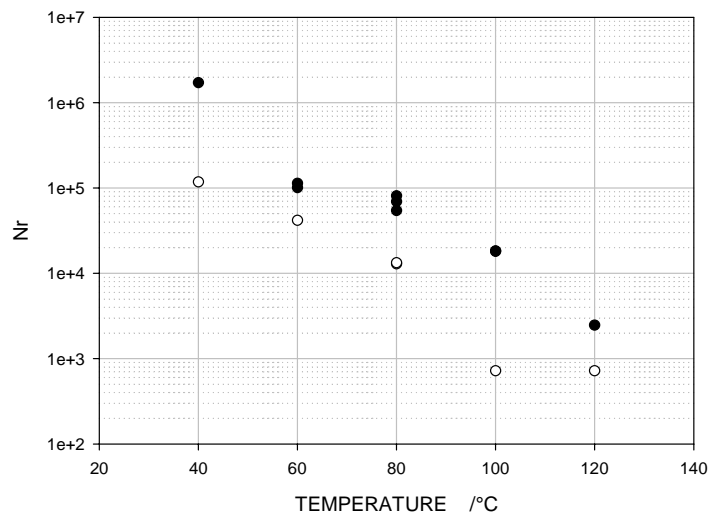


FIGURE 7. Number of cycle for failure versus temperature ($\epsilon_0=10\%$ - $f=30\text{Hz}$) for both elastomers: (●) chloroprene rubber, (○) hydrogenated nitrile butadiene rubber

It turns out that studying crack propagation on DMA is an interesting approach to characterize the fatigue life of a rubber looking at the influence of parameters such as the crack size, the frequency, the temperature.

4. CONCLUSION

The study of fatigue crack growth, with a Dynamic Mechanical Analyser, in CR and HNBR led to the following conclusions:

1. The characterisation of crack propagation of rubber using a DMA shows promising results.
2. For CR and HNBR, the results of our works have shown the influence of defect size on fatigue life.
3. The determination of a threshold – below which the defect has little effect on fatigue life - has been investigated. The present study indicates the existence of a critical defect size, although further studies are needed to confirm this important aspect.

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