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Influence of defects size on the fatigue properties of an industrial EPDM

T. Balutch

Institut de Recherche en Génie Civil et Mécanique (GeM), UMR CNRS 6183, Ecole Centrale de Nantes, Nantes, France

Vibracoustic Nantes, CAE & Durability Prediction Department, Carquefou, France

B. Huneau

Institut de Recherche en Génie Civil et Mécanique (GeM), UMR CNRS 6183, Ecole Centrale de Nantes, Nantes, France

Y. Marco

Institut de Recherche Dupuy de Lôme (IRDLe), UMR CNRS 6027, ENSTA Bretagne, Brest, France

P. Charrier & C. Champy

Vibracoustic Nantes, CAE & Durability Prediction Department, Carquefou, France

ABSTRACT: Fatigue life of rubber components is known to be linked with the defects population. Indeed, cracks initiate around these defects, and propagate until the final failure of parts. In the present work, the defects size influence on the fatigue properties is estimated by drilling calibrated holes of different sizes in pure shear samples and analyzing fatigue crack initiation and propagation stages around these defects. The use of the square root of the area projected on the tensile direction as a representative size measurement allows gathering in the same plot natural defects and calibrated holes results. Finally, it is found that the number of cycles for crack initiation stage is much more influenced by the defects size than the number of cycles for crack propagation stage.

1 INTRODUCTION

Anti-vibration solutions in automotive industry are crucial to ensure comfort, safety and durability in vehicle systems. Most of the parts that are designed in Vibracoustic to provide these services are made of rubbers because of their remarkable mechanical properties such as their damping or their ability to withstand large deformations. Fatigue life of rubbers has been widely studied in literature (Cadwell et al. 1940, Lake and Lindley 1965, Mars and Fatemi 2004) and appears to be strongly connected with defects, which act as crack initiation sites (Abraham et al. 2005, Le Saux et al. 2011, Legorju-Jago 2012, Le Cam et al. 2013, Masquelier 2014, Huneau et al. 2016). Their intrinsic properties, adhesion to the matrix and geometrical features such as shape, orientation and size are assumed to have an effect on the fatigue crack initiation and early stages of fatigue crack propagation. Consequently, different authors have proposed to quantify the defects size influence on the fatigue properties. To do so, two types of procedures exist in the literature: either control and measure the size of the natural defects populations or introduce calibrated defects. For instance, Huneau et al. (2016) proved that the bigger the mean size of carbon black agglomerates is, the shorter the fatigue

life is, by testing five natural rubber filled with different types of carbon black. On the other hand, Abraham et al. (2005) introduced calibrated glass beads of different diameters in the formulation of an EPDM rubber and showed that the bigger the glass beads are, the shorter fatigue lives are.

These results, that can appear intuitive, were known for a long time for metallic materials (Murakami et al. 1989). However, changing the material composition has a complex influence on other parameters and may not only change the defects size. In order to keep a constant material, several authors proposed to machine small defects in metallic fatigue samples, for instance by using drills (Murakami and Endo 1980) or electric discharge machining (Billaudeau et al. 2004). It is then usually shown that below a certain defect size, there is no noticeable influence on the fatigue lifetime results. On the other hand, above this size, fatigue lifetimes constantly decrease as the defect size increases. To our knowledge, this kind of approach has never been tried on rubbers.

In a previous article (Balutch et al. 2018), the damage scenario of the presently studied EPDM rubber is described, and it is shown that each fatigue sample exhibits only one cause of failure. Two types of defects are identified: inclusions, which are

agglomerates of ingredients of the rubbers recipe, and the parting line region which is a mold flaw. A correlation is then established between the parting line size and the duration life, but none is found for inclusions where other parameters must be taken into account as the exact 3D geometry and the position in the sample because of the high strain gradient induced by the hourglass shaped geometry of the samples.

Thus, the aim of this paper is to evaluate in a quantitative manner the defects size influence on the fatigue properties of an industrial EPDM rubber, by introducing calibrated holes in fatigue samples.

2 MATERIALS AND METHODS

2.1 Materials and samples

The material of this study is a fully formulated industrial EPDM, filled with carbon black and cross-linked with sulfur. As it is used for real mass-produced parts, the quantitative formulation is not provided here.

The samples used to perform the fatigue tests are Pure Shear (PS) samples with dimensions 6x40x1 mm (Figure 1). It has to be noted that this sample geometry is usually used for fatigue crack propagation measurements. It is chosen in this study because it is more convenient to introduce calibrated defects in this geometry and to monitor fatigue cracks around them than it is on 3D specimens. In order to be representative of natural defects, the calibrated holes sizes must be in the same order of magnitude, which is approximately 0.1 mm (Balutch et al. 2018). The best way to perform a 1 mm deep defect of such a size is drilling. Consequently, small drills made of titanium alloy (Figure 2), provided by Mikron Tool, are used. The drilling is done in the glassy

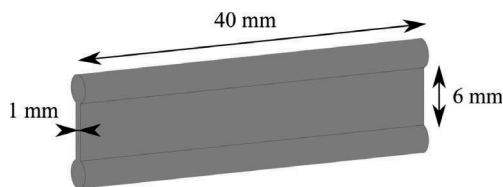


Figure 1. PS sample geometry.

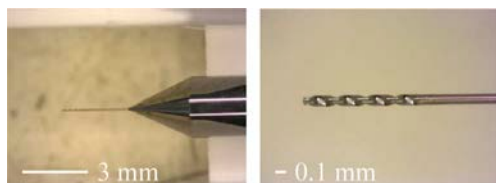


Figure 2. Titanium drill of diameter 0.1 mm.

Table 1. Number of samples for each drill diameter.

ϕ (mm)	0.1	0.2	0.3	0.4	0.5	0.8
Number	7	3	3	4	4	3

state of the studied EPDM rubber, around -70 C, in order not to damage the rubber around the hole. At the end, PS samples have one hole in their center, and the number of specimens corresponding to each hole diameter is reported in Table 1.

2.2 Testing procedure

The fatigue tests are conducted on an electrodynamic machine INSTRON E10000. The mechanical load is displacement controlled, with a load ratio $R = \frac{\Delta_{min}}{\Delta_{max}} = 0$. The maximum displacement is constant for all experiments and set to 12 mm, which corresponds to a macroscopic elongation of $\lambda = 3$. The frequency is set to $f = 10$ Hz, which is the highest possible with such displacement amplitude because of machine limitations.

In order to analyze fatigue crack initiation and propagation around defects, the fatigue test is interrupted every 200 cycles. The sample is then stretched to 3 mm during 2 seconds and a picture is taken. Therefore, a camera is installed in front of the specimen, and is triggered by the tensile machine (Figure 3).

2.3 Observation tools

The camera installed in front of the specimen is a Prosilica GT6600 from Allied Vision with a resolution of 6576*4384 pixels, and a bi-telecentric lens TC16M056 from OPTO engineering is used. The size of the pixel is equal to 8.55, and the distortion is below 0.1 %. The crack detection highly depends on this resolution, and a different experimental device would lead to a slightly different split between crack

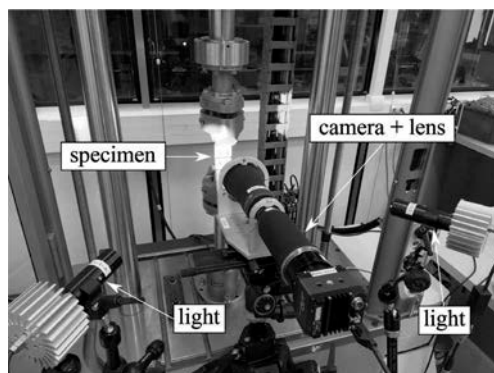


Figure 3. Experimental device.

initiation and crack propagation stages. Indeed, fatigue crack propagation occurs at very small scale and the detection of the crack appearance requires a higher resolution. Furthermore, cracks in the volume or on the unobserved side of the specimen cannot be detected. However, this set-up allows keeping the sample mounted in the fatigue machine which reduces the experiment duration.

Furthermore, Scanning Electron Microscopy (SEM) is used to characterize the quality of the drilling, and to perform fracture surfaces analysis. The microscope is a JEOL JCM 6000plus, the acceleration voltage is set to 15 kV, and the studied EPDM rubber, filled with carbon black, is sufficiently conductive to use secondary electron imaging under a high vacuum.

3 RESULTS AND DISCUSSION

In the first place, it has to be noted that small calibrated defects of good quality are successfully obtained. SEM pictures of all the tested defects sizes are presented in Figure 4. The rubber around the holes seems not burned or dragged by the drilling. This can only be obtained by performing the drilling in the glassy state of the rubber as mentioned in the methods section.

Secondly, a damage description is proposed on Figure 5 thanks to regular pictures taken during fatigue tests. A first fatigue crack initiates on one side of the calibrated defect (Figure 4b), and few cycles later, another crack initiates on the opposite side (Figure 4c). They finally both propagate until final failure occurs. Figure 4d shows the sample just before failure. Each sample exhibits only one root cause of failure.

Duration lives are split between initiation and propagation stages for each fatigue test. The number

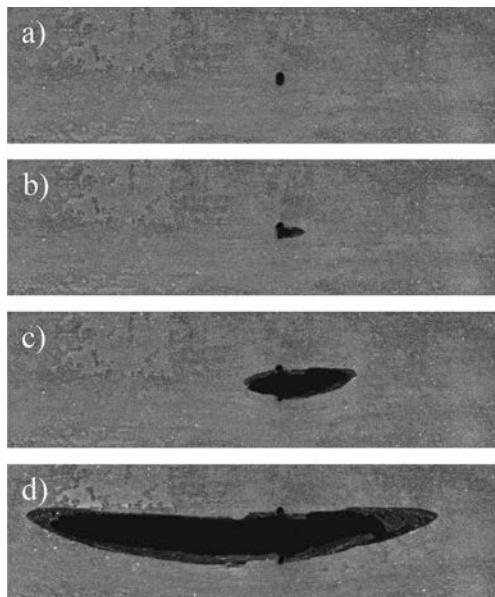


Figure 5. PS sample with a 0.2 mm hole at a) N = 0 cycle b) N = 109 400 cycles c) N = 112 600 cycles and d) N = 116 400 cycles.

of cycles for “crack initiation” is taken as the number of cycles for which a crack is detectable. With the experimental device used, this corresponds to a crack of approximately 1 to 10 pixels, which is some tens of micrometers. The number of cycles for crack propagation is then the difference between total failure and “crack initiation”. The Figure 6 represents the number of cycles for crack propagation as a function of the hole diameter. Only samples which broke on calibrated holes are plotted. The black circles are the mean values of the number of cycles, and the error bars illustrate the scatter for one

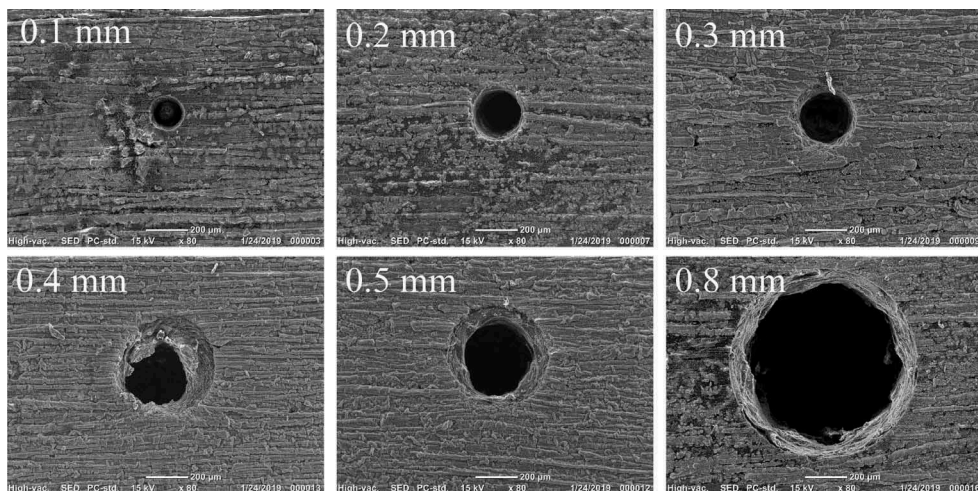


Figure 4. SEM pictures of the different hole sizes at the same magnification (x80).

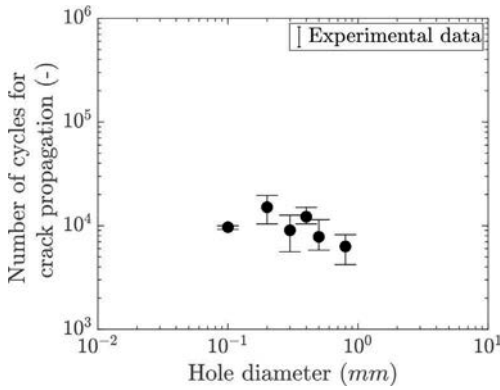


Figure 6. Hole diameter influence on the number of cycles for crack propagation.

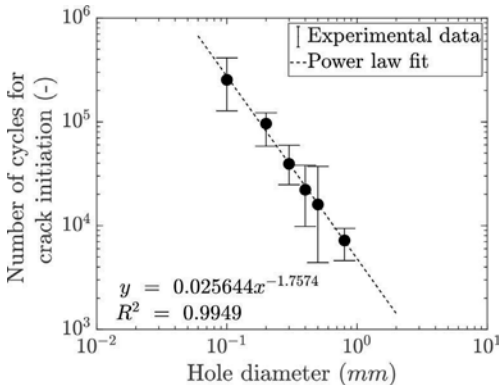


Figure 7. Hole diameter influence on the number of cycles for crack initiation.

given drill diameter. Figure 6 shows that the number of cycles for fatigue crack propagation is almost constant regardless of the hole diameter. In other words, as soon as a crack is visible, the number of cycles to propagate this crack until final failure is not much dependent on the defects size.

Similarly, the Figure 7 represents the number of cycles for “crack initiation” as a function of the hole diameter. Unlike for propagation stage, it appears that the curve follows a decreasing power law with a good correlation coefficient. Thus, the bigger the size of the defect is, the shorter the number of cycles for “crack initiation” is, and this is in accordance with the literature (Huneau et al. 2016, Abraham et al. 2005).

Another point is that Figures 6-7 prove that the total duration life is mainly driven by the fatigue “crack initiation” stage, the crack propagation stage being more or less constant for every samples. The strong influence of the defects size on Figure 6 suggests that the fatigue crack initiation stage must be studied to fully understand the fatigue behaviour of

EPDM. Still, it is reminded that, in this study, the crack initiation stage includes the first stages of crack propagation as mentioned in section 2.3, because of the experimental device used for crack detection.

An additional interesting observation is that, over the 7 samples tested with the hole diameter of 0.1 mm, 3 samples broke because of natural defects. This means that there is a competition between crack initiation on natural defects and crack initiation on the calibrated hole. It is assumed that the most critical defect in the sample in regards to fatigue crack initiation will cause the failure (Balutch et al. 2018). Yet, the criticality of a defect is very complex to evaluate and depends on the type, geometry, chemical nature, interface and internal properties of the defect and the mechanical fields around it.

Considering that geometrical features are the easiest parameters to quantify, the square root of the projected area in the tensile direction of the defect that led to failure is measured for all samples. This allows plotting on the same curve the results for natural defects and calibrated holes. It has been initially proposed by Murakami and Endo (1983), and is still used nowadays in fatigue of metals to represent the defects size (Houria et al. 2015, Le et al. 2016). This area is measured thanks to fracture surfaces analysis (Figure 8) and plotted versus the number of cycles for crack propagation (Figure 8) and the number of cycles for “crack initiation” on the Figure 10. The black points correspond to calibrated holes, and the black circles correspond to natural defects. The Figures 9-10 show that natural defects and calibrated holes seem aligned on one master curve. It appears that the defects size has a minor influence on crack propagation stage compare to crack initiation stage. Furthermore, Figure 9 shows that a variation of 1 decade in the \sqrt{area} of the defect responsible of the failure roughly leads to a variation of 2 decades in the number of cycles for fatigue crack initiation. Regarding the fatigue results of EPDM (Flamm et al. 2008, Balutch et al. 2018), it is common to observe more than 2 decades of scatter in the fatigue lifetime results. Hence, the variation of the defects size could explain a part of the scatter in the fatigue lifetime of such material.

This means that \sqrt{area} of the defect could be a good criterion to compare the defects size criticality. However, it must be reminded that these results are obtained for one load level only, and with a specific sample geometry. Furthermore, the validity of \sqrt{area} as representative of the defect size is questionable. Indeed, fatigue crack initiation is a local phenomenon and perhaps only a localized zone of the defect contributes to this stage. Moreover, two defects could have the exact same projected area with very different 3D shape, and a lot of other parameters previously mentioned could have a noticeable influence on the fatigue crack initiation and propagation stages. For all of these reasons, the use of \sqrt{area} requires further investigations.

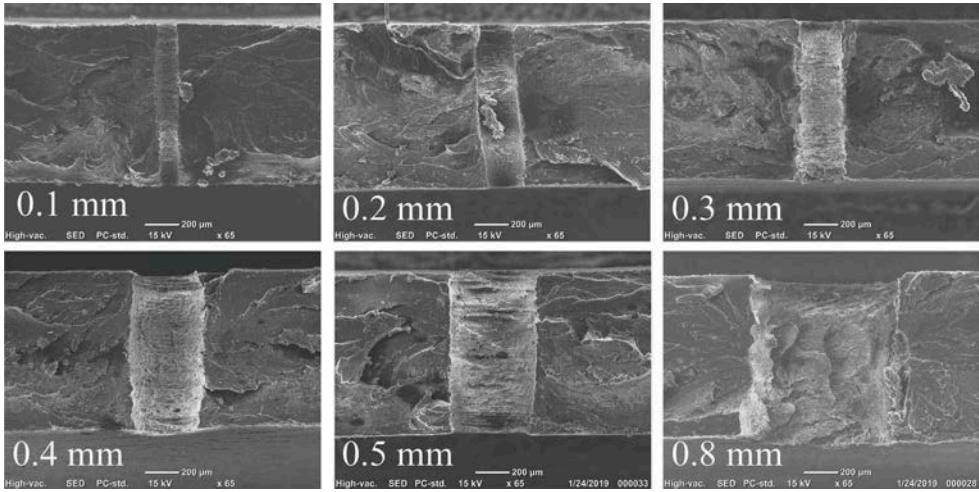


Figure 8. SEM pictures of fracture surfaces for each hole size at the same magnification (x65).

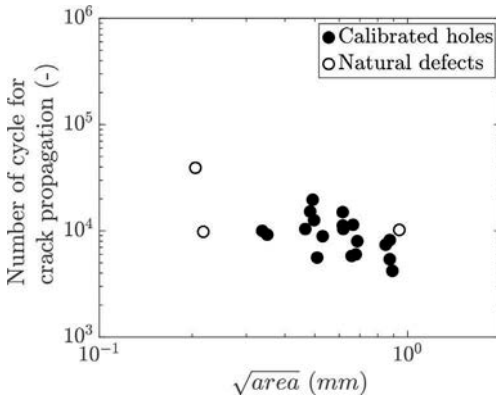


Figure 9. Defect $\sqrt{\text{area}}$ influence on the number of cycles for fatigue crack propagation.

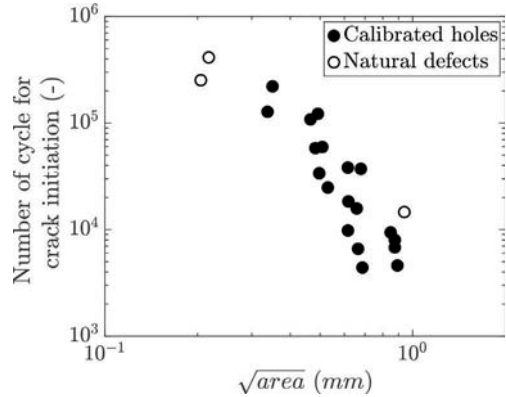


Figure 10. Defect $\sqrt{\text{area}}$ influence on the number of cycles for fatigue crack initiation.

Finally, in the literature concerning the fatigue of metals described in the introduction (Murakami and Endo 1980, Murakami et al. 1989, Billaudeau et al. 2004), it is shown that defects size have no influence anymore on the fatigue lifetimes below a certain size. The results obtained for the studied EPDM in Figure 10 only confirm an increasing duration life with decreasing size of defect. But no assumption can be made on how defects with $\sqrt{\text{area}}$ below 200 would influence the fatigue resistance. Also, a fundamental difference between rubbers and most of metallic materials is the absence of fatigue limit for rubbers, which could explain the absence of thresholds in the fatigue properties of rubbers. The possible existence of a plateau or the continuation of the slope on the left hand part of the Figure 9 remain open questions. In fact, the influence of these small defects sizes is very hard to investigate. Indeed, for

PS samples with holes of diameter 0.1 mm, 3 out of 7 broke on natural defects. It therefore appears that the criticality of this size of calibrated hole is close to the criticality of natural defects. Consequently, if calibrated holes with diameter smaller than 0.1 mm are introduced, it is very likely that natural defects will cause the samples failure. Then, it could be inaccurately concluded that below 0.1 mm, there is no more influence of the defects size on the fatigue lifetime. But actually, this would only be due to the fact that calibrated holes are no longer the cause of failure. This analysis suggests that the range of holes diameters investigated in this study is relevant for the material considered. This also confirms the importance of fracture surfaces analysis to identify the real cause of failure in order to improve the understanding of the defects size influence on the fatigue properties.

4 CONCLUSIONS

The present investigation proposes a method to evaluate in a quantitative manner the influence of the defects size on the fatigue properties for a given rubber formulation. To do so, calibrated holes of sizes comparable with the natural defects sizes are introduced in PS samples. These latter are then subjected to fatigue tests and regular observations are performed.

The fatigue “crack initiation” and propagation stages are split thanks to the detection of a crack of a few tens of micrometers. The results show that the number of cycles for crack propagation is more or less constant for every sample and approximately equal to 10^4 cycles for a given nominal load level. This shows that the propagation stage is not much influenced by the defects size. In the contrary, the defects size has a strong effect on the number of cycles for “crack initiation”, and a power law fit allows quantifying the results. The square root of the projected area of the critical defects on the tensile direction is then measured thanks to fracture surfaces analysis. This parameter allows gathering on the same curve the results for calibrated holes and natural defects. It appears that fatigue lifetimes, for an initiation criterion, constantly increase with decreasing defects size, but no assumption can be made on the influence of defects with \sqrt{area} smaller than 200.

To conclude, this study proves that the defects size has a major influence on the fatigue crack initiation stage for EPDM materials, and that the crack initiation, including the first stages of crack propagation, could be the key parameter to fully understand the fatigue behavior of EPDM. Moreover, knowing that there is always only one defect driving EPDM failure (Balutch et al. 2018), the scatter in defects size, among others, could explain a part of the scatter in the fatigue results of such materials.

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