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Research Paper

Influence of combined carbon-epoxy on the fatigue life of elastomer materials

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ABSTRACT

The Rubber-carbon black composite material-based product is the main matrix used in domestic and industrial applications in the last decades. This study proposes replacing the reinforcing carbon with matrix-neutral rubber with epoxy resins to reduce the carbon footprint and lower the environmental impact while enhancing the product's mechanical properties. Five additive percentages of carbon–epoxy, starting from 100-50% Carbon-black and 0-50% Epoxy, were studied using a hybrid experimental-numerical approach. Experimentally, the extension fatigue, flexural fatigue, and elongation modulus testing were completed using the universal testing machines with a low loading rate (quasi-static process). Numerical, the finite element analysis by the commercial software ABAQUS-SAE within the built-in hyperelastic constitutive model was utilized to visualize the full-field stresses. The experimental nominal stress-strain data was used as input for the numerical model, and Ogden's formula was applied to simulate the mechanical response of the specimens. The stress field and stress concentration of different experimental tests are given. The effect of increasing the additive epoxy on the rubber-carbon mechanical response and the fatigue life under repeated loads were identified and discussed. Furthermore, the results show that a small amount of epoxy can be used as a reinforcing material for a rubber compound and it improves the mechanical properties. This along with more results are shown in the result section.

1. Introduction

In the last decades, many researchers have studied rubber and its composites because of its importance and wide use in different fields. Nowadays, rubber composite has been applied in many new and industrial applications due to its good mechanical characteristics and ability to sustain significant stress without any permanent deformation or fracture. The compounds of rubber composite have been used as a protector in several technical apparatuses, bridges, and buildings against the effects of vibrations due to their high elastic property [1–3]. The cost-effective material makes them ideal for many applications including bushings, tie bars, and nozzle ports, which should endure the distortion generated by the heat deformation of the exhausts[4]. On the other side, black carbon has a direct impact on the environment and climate change by discharging heat energy into the atmosphere [5–7]. Rubber compounds are subjected to multiple stresses and factors that affect the fatigue life due to repeated stresses during the operation, so it is necessary to investigate its mechanical properties and fatigue life to understand the mechanism of failure and how it effectively develops in the material. Rubber components have been subjected to varying loads that frequently fail because of the nucleation and propagation of the cracks [8, 9]. Epoxy resin is one of the thermoset material types that harden when exposed to energy (thermal, chemical reaction, or radiation). When it is mixed with a catalyst or hardener, it forms an insulating layer characterized by hardness, adhesion, weathering, and resistance to friction, so it is often used as paint or adhesive [10]. The Epoxy groups are combined with the hardener and mixed to react, forming large, forked particles, and the size of the particles increases with the continuation of the cure. Curing agents (fillers) must be added to obtain new epoxy aggregates because it is an important part of the treated compound, which gives the final properties of the mixture [11–15].

Fatigue is a type of mechanical failure that happens in materials when they are subjected to a periodic load (as in speed bumps, bearings, tires, gaskets, bushings, hoses, belts, seals, and mounts), which would not cause the material to fail if applied statically when its value is less than the value of the yield stress [16]. In general, the fatigue failure process is divided into three main stages. The first stage is crack nucleation, which is connected to the limit of endurance, whereas no cracks are initiated if the input of the energy is less than the endurance limit. The second stage is caused by an energy input that exceeds the limit of endurance, which causes the growth of nucleated cracks. The deterioration of the rubber substance continues in the last phase. The formation of a crack, its development, and finally its overall failure are all considered factors explaining the mechanics of fracture behavior of rubber under fatigue loading circumstances [17, 18]. Fatigue rate is commonly described as a cycle number required to reach a specimen into the failure at specific stress for stress-controlled tests or a particular strain for strain-controlled testing. The experimental data on fatigue life are dispersed. In time-consuming tests, many samples are usually required to get an average value of rubber fatigue life [19, 20]. Therefore, experimentally defined fatigue fracture behavior as the fatigue crack growth (FCG), rate function, (R) (it is the increase in the crack growth (da) when the number of cycles (dN) increases) [21, 22]. This work proposed using epoxy, Eco-friendly, as a filler material instead of black carbon. The epoxy was added to the rubber by reducing the amount of black carbon gradually and investigating mechanical properties and the best filler percentage that could be used. This paper is organized as follows: the experimental setup and the specimen preparation are presented in section 2. Section 3 includes the numerical analysis using the ABAQUS program. Followed by the result and full field stress in section 4. Finally, the conclusion and recommendation are in Section 5.

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Nomenclature:

Greek Symbols:		**Continued Greek Symbols:**	
σ_{xx}	Normal Stress along x-axis	R_x	Rotation around x-axis
σ_{yy}	Normal Stress along y-axis	R_y	Rotation around y-axis
σ_{xy}	Shear Stress in xy-plane	u	Displacement along x-axis
σ_e	Von Mises stress, (Equivalent stress)	v	Displacement along y-axis

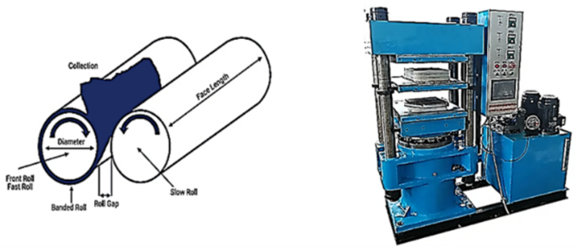
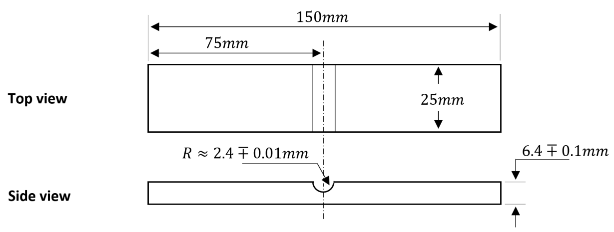
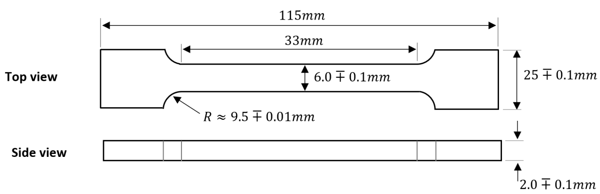


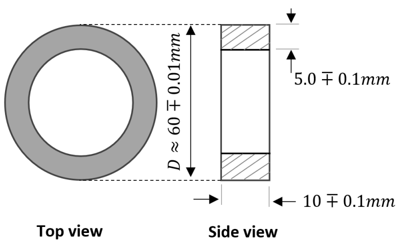
Figure 1. Experimental facilities used in this work, (a) The rolling machine; (b) The thermal press machine.



(a) Fatigue specimen



(b) Tensile specimen



(c) O-ring specimen

Figure 2. Schematic of the specimens used in this test where (a) The fatigue test specimen, (b) The tensile test specimen, and (c) the O-ring modulus specimen.

2. Experimental Program

2.1 Material manufacturing details

The rubber served as the matrix material, and carbon black, and epoxy resin served as the reinforcing elements. The practical aspect was implemented in follows stages. *First*: The rubber material was supplied by the State Company for Rubber and Tires Industry, (SCRTI), in Diwaniyah, Iraq. The basic rubber paste components are listed in Table 1 below.

Second: The epoxy utilized in this project is called $Sikadur-52$, and it is made up of two high-grade components, low-viscosity $(1000-250\ MPa/s)$, yellow in color, has a density of around $(1.1\ kg/l)$ at $20\ C^{\circ}$, and have a weight-based mixing ratio of (2R:1H). The epoxy resin and the hardener were manually combined in a (2R:1H) ratio at room temperature $25C^{\circ}$. To be homogeneous and free from air bubbles, the mixture of epoxy was added gradually to the natural rubber dough in varying amounts of the rubber weight $(10-50\%)$.

2.2 Specimens preparation

Three different types of specimens were prepared, the bend flexing specimen with circular groove for the fatigue test, the dog-bone tensile test specimen for the uniaxial tensile test, and a circular ring for the elastic modulus test. Six groups of doughs were prepared each one with a different amount of epoxy as 0% (No-added), 10%, 20%, 30%, 40%, and 50% of epoxy respectively. The dough was inserted into the rolled machine to get a uniform sheet, Fig. 1a. Equipment has been employed to regulate the space between the two rollers so that it can range from a minimum of 2 *mm* to a maximum of 8 *mm*. A unique mold was used in the thermal press machines, Fig. 1b, to press the dough into the final specimen shape under the ideal conditions of 3.5 *MPa* pressure and 146 C°temperature for 15 to 20 minutes to produce vulcanized composite rubber.

2.3 Mechanical testing

2.3.1 Flexing (Bending) fatigue test

Eighteen standard specimens were prepared for the fatigue test according to the ASTM-D430 [23]. As shown in Fig. 2a, the sample has a total length of 150 *mm* and a width is 25 *mm*. At the center, there is a small circular groove with a radius of 2.39 *mm*, which is important for the stress concentration that was used, [24]. The specimens are held from both sides in the fatigue testing machine, Fig. 3. One side is fixed and the other end is moving. A fatigue test has been carried out for the manufactured samples using the De Mattia Flexing Machine Test type F16 fatigue test device. Samples have been fixed in special clamps in the device, so they can flex and tensile during the test time, Fig. 3. The central carriage moves up and down to flex the samples during operation at a certain temperature.

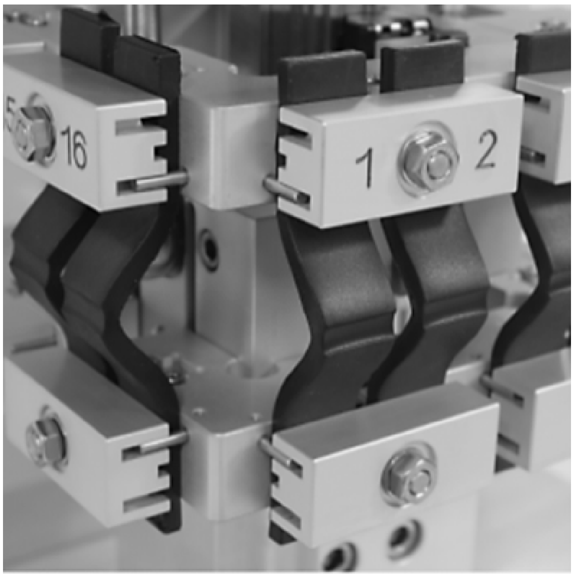


Figure 3. Fatigue testing device (De Mattia Flexing Machine).

2.3.2 Extension fatigue test

In this test, eighteen samples were prepared according to specifications ASTM D412 [25], Fig. 2b. The specimen has a total length of 115 *mm* with a thickness of 2 *mm*. The gauge length is 33 *mm* and the gauge width is 6 *mm*. The specimens were fixed at specific distances to ensure that periodic expansion stress was applied. The sample in the same way, the growth and progression of the scratch of the samples are monitored and the information is fixed, see [12, 26]. From the two tests, the life of the specimen with different epoxy weights has been examined.

Table 1. The elements components of the Rubber dough matrix supplied by SCRTI.

	Element	**Occupation**	**Mass weight** (%)
Matrix	Rubber RSS1	Basic material	≈ 60
Black carbon	N_{110}	Reinforcement filling	≈ 28
	N_{326}	Reinforcement filling	
Supporting elements	Sulfur	Vulcanization agent	≈ 12
	Renacit	Digestive substance	
	Stearic acid	Tonic	
	Zinc oxide	Tonic	
	DCBS Accelerator	Accelerated	
	TBBS Accelerator	Accelerated	
	PVI	Cathodic substance	
	Escorez 1102	Adhesive	
	Resorcinol melt	Adhesive	
	IPPD	Oxidation protection	
	Wax	Oxidation protection	
	Struktol 40 ms	Homogenizing substance	
	HMT	Adhesive	

Table 2. Finite element model details.

Model	**No. of Node**	**No. Elements**	**Type**	**Size**
Tensile	23676	18705	C3D8RH	$\approx 250\mu m$
Fatigue	25978	22560	C3D8RH	$\approx 300\mu m$

Table 3. Maximum tensile strength of the rubber epoxy materials.

Epoxy additive, (%)	0	10	20	30	40	50
Max. Strength, (MPa)	12.387	10.997	08.662	12.081	09.619	06.259

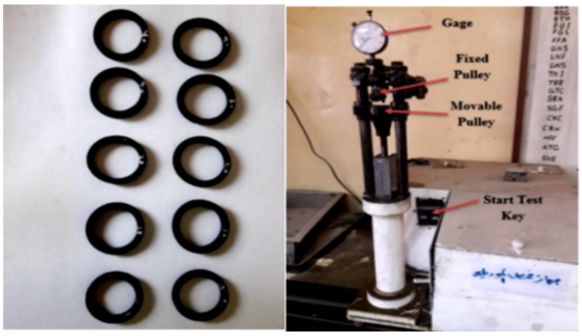


Figure 4. Specimens' preparation and testing machine (a) Modulus test samples, (b)Modulus test device.

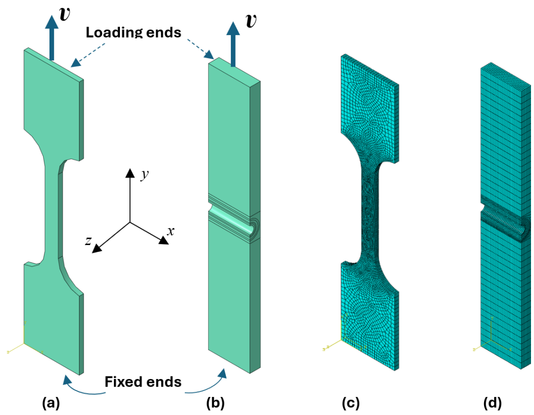


Figure 5. The CAD and FE model of the Tensile and fatigue specimens. Where a) the dog-bone tensile specimen, b) the rectangular with semicircle grove specimens, c, and d) the mush model of the tensile and fatigue specimens respectively.

2.3.3 Modulus test

This test was carried out according to the ASTM D1414 to estimate the modules of the rubber composite [3]. Where the samples were made with a special model and then cut into rings, Fig. 4. The rings had an inside diameter of 50 *mm* a thickness of 5 *mm* and a width of 10 *mm*. The specimen is installed in circular jaws where it can be bulled up to the failure. To avoid repetition, the experimental data was used as input to the analytical solution that is available in ASTM D1414 to calculate the modulus of the rubber and the audience could see [27] for analytical details.

3. Numerical analysis

The finite element analysis of the tensile and fatigue specimens is used to investigate the stress components under uniaxial load conditions. Thus, the ABAQUS/CAE software is utilized as follows:
The CAD models (tensile and fatigue) are created by CAE tools and more details can be found in [28–30]. The Dog-Bone specimen, Fig. 5a, for the tensile test with the standard dimension regard to the ASTM D430. In the case of the fatigue specimen, Fig. 5b, the rectangular plate 150256.35 *mm* with a semicircle groove with a radius of $r_o \approx 2.39$ *mm*, is used to concentrate on the tearing location. The CAD models are meshed with the linear hexahedral elements C3D8RH, Fig. 5c, and d, which is a 3D linear brick continuing 8-node with hourglass control. More details are listed in Table 2.

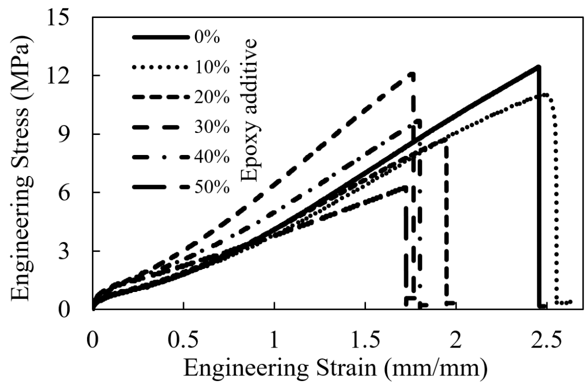
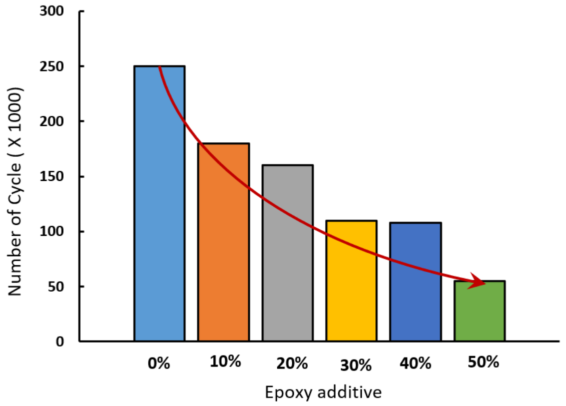
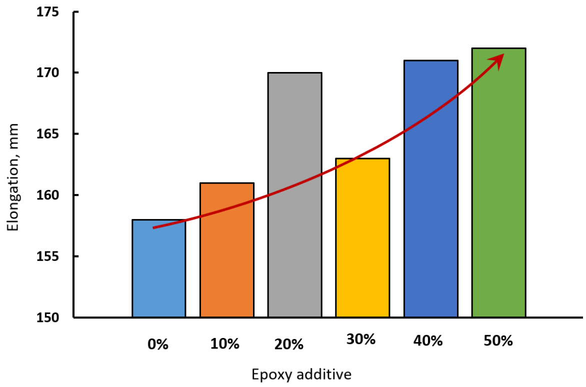


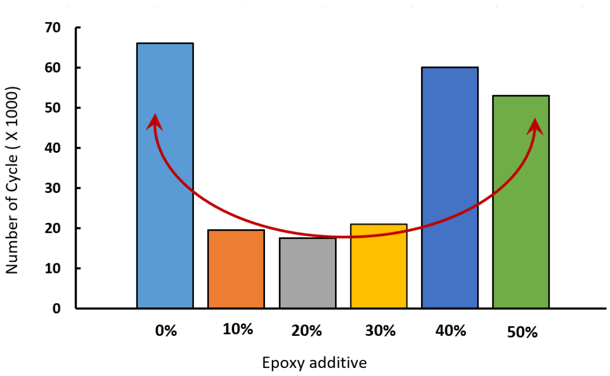
Figure 6. Typical experimental results of the stress-strain of the rubber epoxy with different additive amounts according to the ASTM D412.



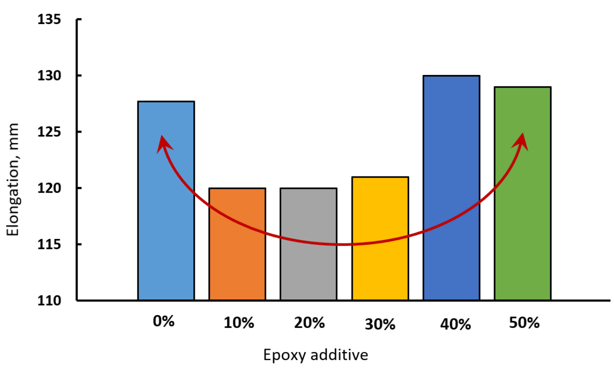
(a) Bending test



(b) Bending test



(c) Extension test



(d) Extension test

Figure 7. The effect of epoxy additive ratio on the fatigue life and elongation of the rubber from two different tests. Where a) Fatigue life from the bending test, b) Elongation of rubber from the bending test, c) Fatigue life from the extension test, d) The elongation from the extension fatigue test.

Two types of boundary conditions were applied to the specimens. First, one end is fixed in all directions, $u = v = R_x = R_y = 0$. Second. The other end is subjected to the displacement along the y-axis, $u = R_x = R_y = 0$. The hyperelastic behavior of rubble was modeled depending on the strain-energy potential of the Ogden model. The initial shear modulus and bulk modulus used in the Ogden strain formula were calculated from the experimental measurements of nominal stress and strain. data. The numerically, full-field stress (contours) along the transverse direction, (σ_x), longitudinal direction (σ_y) , and in-plane shear stress (σ_{xy}) are presented in the result section. Furthermore, the von Mises stress is presented to predict the yielding locations of rubber specimens under complex loading developed from the uniaxial tensile tests.

4. Results and discussion

The fatigue life and crack initiation effectively give a good indication of the improvement and effect of additive materials on the base matrix materials. The natural rubber composites combined with epoxy at various percentages, including (0%, 10%, 20%, 30%, 40%, and 50%) from carbon black, have been tested in several experiments, and the findings have been compared with samples of 0% epoxy natural rubber. The engineering stress-strain of different epoxy additive ratios is shown in Fig. 6. As shown in Fig. 6, for all stress-strain data the rubber is not linear special at the strain less than 1. The rubber shows a higher modulus at the strain less than ≈ 0.2, at the strain from 0.3 up to ≈ 1 the modulus is less than ≈ 3 MPa, the modulus increases again up to the failure. This behavior is shown with all presented epoxy additives. The epoxy effect directly on the maximum strength of the composite. At 0% of epoxy the maximum strength reaches 12.3 MPa, while at 20% of epoxy, the strength is about 8.66 MPa. With more epoxy up to 50%, the strength decreases to 6.259 MPa as shown in Table 3. From Table 3 and Fig. 6, the best percentage of epoxy to improve the modulus and strength is 30% of rubber.

4.1 Fatigue life under bending and extension test

The fatigue life data for both bending fatigue, and tensile fatigue, show a different cyclic life presented related to different loading directions. As shown in Figs. 7a and 7c, the number of life cycles decreases with an increase in epoxy from 250,000 to 55,000 cycles, under the out-of-plane fatigue load (Bending fatigue). In the case of in-plane fatigue load, and tensile fatigue, the number of cycles decreases then jumps up as the epoxy rate increases, Fig. 7c. Overall, the bending fatigue shows a decay exponential function. In contrast, the tensile fatigue presents the U-shape. The maximum extension of bending and the tensile is shown in Figs. 7b and 7d, respectively. The elongation from the out-of-plane test shows clear proof as the stretch from 160 mm up to 172 mm as the epoxy continues increase, however, the elongation test from the tensile test has a U-shape relation to the epoxy, Fig. 7d. Thus, for the engineering application required good elongation and bending load, the 20% of epoxy is a good choose, however for the applications under tensile load and longer life as well as good elongation, the 40% of epoxy is recommended. In general, Fig. 7, illustrates the comparison between the standard rubber-black carbon to rubber-carbon-epoxy added in different ratios. The fatigue life decreases with an increase in the epoxy unless the tensile shows an increase at 40 and 50% of epoxy. Furthermore, the elongation of samples containing epoxy compared to natural rubber increases since the epoxy improves the softness properties of rubber.
The crack growth during the bending test and tensile test are presented in Fig. 8 and Fig. 9, respectively. In the case of out-of-plane load, Fig. 8, with increasing epoxy content in natural rubber, crack growth is slower because the epoxy has more ability to absorb elastic energy than the black carbon. The seed of damage is initiated at the surface of the rubber then more density of damage develops to crack growth. On base materials with 0% epoxy, the crack increases suddenly from 5 mm to 25 mm, while the rubber with 10% epoxy, the crack propagates slowly up to 17 mm and then jumps to the filar. With a 20% epoxy the crack growth slowly up to 20 mm. As the epoxy increases to 30%, 40%, and 50% the crack growth decreases again and the maximum growth is about 15 mm. Thus, the best ratio of epoxy to reduce the filler epoxy is 20% in case of out-of-plane loading.On the other side, when subjected to in-plane loading, the base carbon black rubber cracking grows to a magnitude of 1 mm before rapidly growing into a full crack and then failing. By adding 10% epoxy, the crack gains higher stiffness and can progress to a length of 3 mm before reaching failure. Epoxy ratios of 20% and 30% showed no change in crack growth. The 50% epoxy presents more staple crack growth up to 5 mm before the specimen field. However, the 40% epoxy percentage increases the fatigue life of the rubber and crack growth stability as well. The 10% epoxy is recommended for the specimen subjected to the tensile load because the crack propagation is more stable with not much negative effect on the mechanical

properties.

4.2 Modulus prediction with O-ring rubber test

Figure 10 shows the relationship between the epoxy ratios and tensile modulus from the o-ring test, ASTM D1414. The modulus increases when increasing the epoxy, because of the high crosslinking density between epoxy molecules, the long polymeric chains, and its high resistance to deformation. Due to its small granular size, has a more homogeneous distribution and better penetration, which provides an ideal impediment to the movement of rubber chains and reduces the free volume within the material. When subjected to tensile stress, the rubbers strengthened each other, resulting in a greater tensile modulus in the composite. The 20% of epoxy added to the rubber is enough to improve the modulus by 30%.

4.3 Numerical validation and full field stress

The numerical non-linear analysis of rubber specimens with zero epoxy ratio of tensile and fatigue specimens' model was treated by ABAQUS. The displacement and stress contours are presented in Fig. 11 up to Fig. 14, respectively. As shown in Fig. 11a and Fig. 11b, for both tests, the specimens deform uniformly starting from zero at the bottom end up to the maximum displacement at the upper end v= 7.48 mm. Figure 12a and Fig. 12b, show the stress observed while the tensile rubber specimen was stretched up to 7.48 mm. At this moment, the transverse stress reached a maximum of ≈ 49 KPa in tension and ≈ 13.6 KPa in compressive, while the longitudinal stress reached the maximum tensile of ≈ 251.9 KPa and in compressive is ≈ 590 Pa.

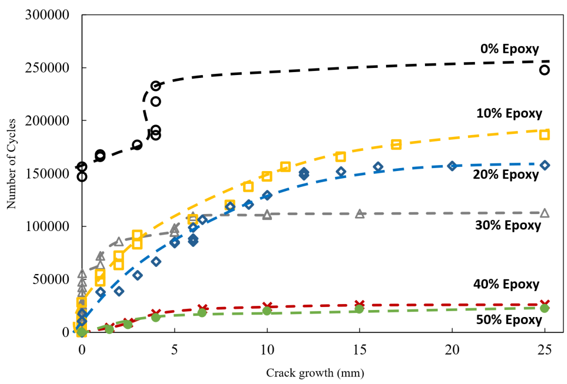


Figure 8. The fatigue life and the length of the crack under the bending test

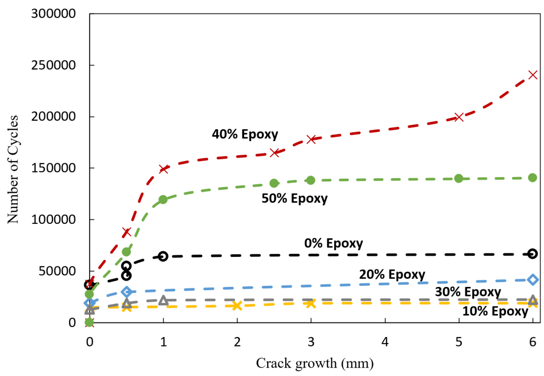


Figure 9. The fatigue life and crack growth of the specimen subjected to tensile periodic load

The maximum stress in the transverse case is developed at the transition zone from the ends to the gauge section, while the maximum stress in the longitudinal direction is at the middle of the gauge. Furthermore, the shear stress of the dog-bone rubber specimen is shown in Fig. 12c. The shear stress is symmetric around the vertical line, and the maximum shear develops at the locations where stress concentrates (corners). The Von-mises stress (equivalent stress) σ_e shows uniform distribution along the gauge length under the uniaxial tensile test. In general, modeling the tensile test with the experimental data as input shows excellent matching between the experimental results and analytical predictions of hyperelastic materials.The fatigue specimen was investigated numerically under uniaxial tensile load as well to verify the experimental result. Thus, at the same extension level, the longitudinal and transverse stress developed at the semicircle groove is increased significantly by 3X and 2X respectively, Fig. 13a and Fig. 13b. However, the shear stress at the groove is less than that in the tensile specimens, i.e. approximately $(0.5X)$. Thus, the semicircle shape reduces the effect of shear stress and the specimen damage under pure tensile stress as shown in Fig. 13c. The Von-misses stress flow around the groove is symmetric and the maximum value is almost at the center, Fig. 14. Also, as the extension field increases up to 10 mm, Fig. 14, the Von-mises increases up to 1.5 MPa and the specimen is mostly damaged at the deepest point of the groove. In general, adding a semicircle groove to the fatigue specimen reduces the effect of shear stress, and the specimen is tested under the uniaxial loading condition as required.

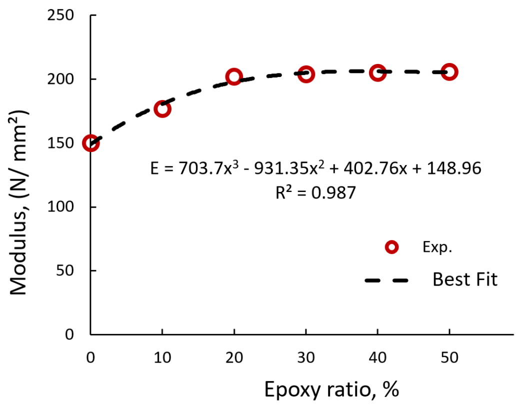


Figure 10. The typical relationship between the modulus of rubber epoxy specimens vs epoxy ratio.

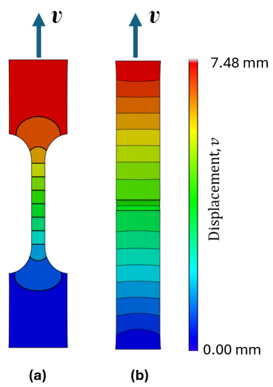


Figure 11. A typical displacement profile of testing rubber with zero epoxy ratio, where a) Tensile specimen, b) Fatigue specimen.

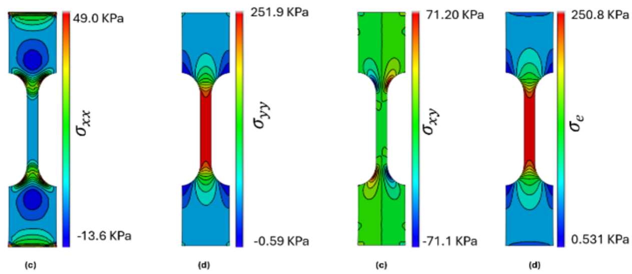


Figure 12. Typical full-field stress of the rubber specimen with zero epoxy ratio subjected to uniaxial extension. Where: a) The transverse stress σ_x ; b) The longitudinal stress σ_y ; c) The shear stress σ_{xy} ; and d) The Von-mises stress, equivalent stress σ_e.

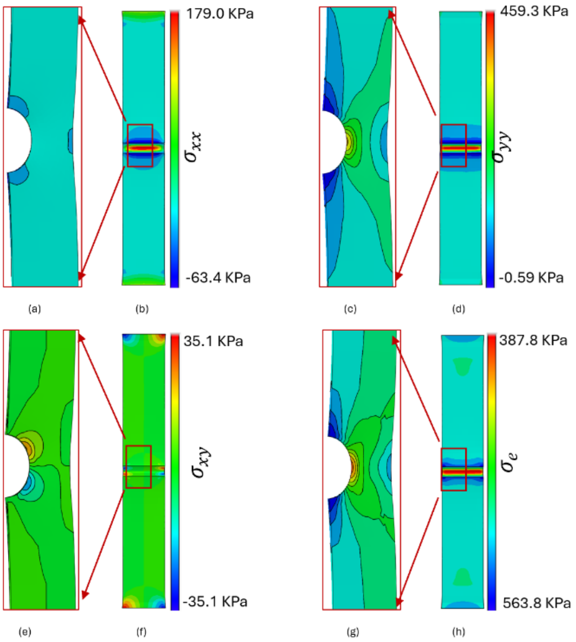


Figure 13**.** Typical Full-field stress of the rubber fatigue specimen with zero epoxy ratio subjected to uniaxial extension. Where: a, b) The transverse stress σ_x ; c, d) The longitudinal stress σ_y ; e, f) The shear stress σ_{xy} ; and g, h) The Von-mises stress, equivalent stress.

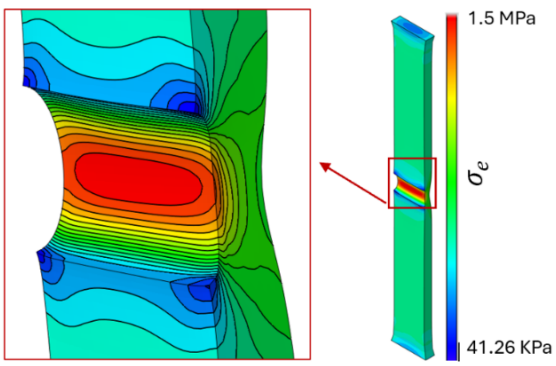


Figure 14**.** Typical Von-mises stress distribution of fatigue specimen subjected to 10mm displacement along the longitudinal direction

5. Conclusions

The novel rubber-black carbon-epoxy material is proposed, and its fatigue life is investigated under out-of-plane and in-plane loading conditions. The RSS1 material contains rubber mixed with carbon and another element. The epoxy was added as 10%, 20%, 30%, 40%, and 50% of the rubber weight. The epoxy is used to reduce the amount of black carbon, increase the use of Eco-friendly materials, and modify the mechanical properties. The experimental-numerical hybrid test showed that the fatigue life of the new rubber composite decreased significantly as the percentage of epoxy increased during the out-of-plane loading condition. However, in the case of extinction, the materials showed an increase in stretch ratio. The tensile-fatigue specimens have also been numerically investigated under uniaxial tensile load to verify the experimental result at the specific extension level. The following are the main concluding points:

- In the case of bending loading conditions, for high performance, no epoxy needs to be added.
- In the case of bending and good extinction material required, 20% Epoxy is advised.
- In the case of tensile loading conditions required with high extinction, 40% epoxy is recommended.
- For more energy absorption and good modulus, 20% of epoxy is suggested.
- To increase the crack growth stability, 10% and 30% of epoxy are advised for bending and tensile load conditions respectively.

Authors' contribution

All authors contributed equally to the preparation of this article.

Declaration of competing interest

The authors declare no conflicts of interest.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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