

A MICRO-MECHANICAL CONSTITUTIVE MODEL TO PREDICT HYGROTHERMAL AGING OF CROSS-LINKED POLYMERS

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ABSTRACT

A multi-physics material model is presented to describe the effects of temperature, oxygen, and humidity on the constitutive response of cross-linked polymers. The effect of hygrothermal damage on the mechanical integrity of the polymer matrix can be considered as the result of damage accumulation of two independent aging mechanism namely, i) thermo-oxidative, and ii) hydrolytic aging. In order to capture the mutual effects of thermo-oxidative and hydrolytic aging, an assumption has been made that each of the aging phenomenon can be superposed to each other. In fact, each of them works independently and as a result, they can compete with each other. Utilizing the theory of network decomposition, all phenomena and their correlation were modeled and thus, the strain energy function of the polymer matrix is written with respect to four independent mechanisms, i) the shrinking original matrix that has neither been attacked by water nor oxygen, ii) conversion of the first network to two new network due to the reduction and formation of cross-links, and iii) energy loss from network degradation due to attack of the water molecules to polymer active agents. Moreover, the pro-

posed model is micro-mechanically based and is mainly relevant on thin samples due to our underlying assumption of homogeneous diffusion of oxygen and water throughout the matrix. The model has been validated against extensive data-sets obtained from experiments we specifically designed for concept validation.

1 INTRODUCTION

In view of their inimitable properties, cross-linked polymers have become widely used in many sensitive applications. They are nowadays used in the auto industry e.g. for vibration damping bearings in car manufacturing, shipbuilding and offshore applications, and energy storage [1–8]. Furthermore, in-service, parts made of cross-linked polymers are often exposed to high temperatures while undergoing cyclic loads. The design of such components is a challenging task since the constitutive behavior of cross-linked polymers changes when they are exposed to harsh environmental conditions for a long time, a process generally known as aging. The aging process, which is often considered as a gradual alteration in the mechanical properties of a component, can be formed due to an individual or combined effects of dif-

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ferent degradation processes. Thermo-oxidation [9–15], hydrolysis [16, 17], hygrothermal [18, 19] and photo-oxidation [20–22] are some of the main types of chemical aging in which oxygen, water, and UV play major roles in chemical aging, respectively. Such classes can be either result of individual aging elements or due to combined synergized effects of the above mentioned environmental factors [23]. Modeling the behavior of polymeric materials against different categories of chemical aging is the center of attention for decades [24, 25]. Any type of chemical aging can induce irreversible damages to the polymer matrix, results in the reduction of materials nominal service life. While predicting any of these types of aging is a great interest of researchers in the last few years, there is still a long way to find a comprehensive model which can consider the synergized effects of chemical aging.

So far, we have studied the individual effect of hydrolytic [26, 27] and thermo-oxidative aging [28] and the contribution of their decay functions on the constitutive behavior of cross-linked polymers in our recent works. Following the fundamental formulations presented in those studies, our goal here is to provide a newly physically-based model to predict the constitutive behavior of cross-linked polymers exposed to hygrothermal aging which is assumed as the combination of thermal and hydrolytic aging. Due to the synergistic effects of combined hydrolytic, thermo-oxidative, and hygrothermal aging, the devised model should be able to capture the constitutive behavior of polymers in all three mentioned aging scenarios simultaneously.

Aging is a complex continuous phenomenon that involves several chemo-mechanical aspects of the material. While testing in the real-time aging trajectory can be expensive and challenging, the experimental investigation of this phenomenon is among key interests of the mechanical engineering community. Therefore, several experimental studies were conducted to investigate the behavior of the polymeric components along with the solo chemical aging mechanisms. Accordingly, Celina et al. [29, 30] comprehensively reviewed the accelerated testing methods while Gillen et al. [31] mentioned the potential deficiency of these approaches. Moreover, several efforts have been done to find the effect of thermo-oxidative aging on the viscoelastic response of the polymers [32, 33]. Similarly, Pazur et al. [34] studied the bulk properties of rubber during thermal degradation. Moreover, Slater et al. [35] conducted experimental research to find the effects of different conditions (i.e. thermal, mechanical, and humidity) on the mechanical performance of thermo-plastic polyurethanes. They observed that the presence of water causes plasticizing of the polymer matrix and results in a greater amount of compression set. Ensuing similar trend, Farrar and Gillson [36] performed a series of experiments on polyglyconate-B to find the relation between polymer morphology and degradation rate. Besides, Rodriguez et al. [37] employed analytical experiments on polyethylene to understand the transient strengthening behavior under photo-oxidation aging. Although the popularity of the models to predict the behavior of materials during

aging is increasing, aside from our previous models [26, 28, 38], the theoretical efforts for that are limited. In this respect, Ha-Anh and Vu-Khanh [39] established a phenomenological model based on the Arrhenius function to predict the behavior of polychloroprene rubber during thermo-oxidative aging. Moreover, Johlitz et al. [40] presented a physically-based model to simulate the mechanical response of automobile bearings during thermo-oxidative aging. Ha-Anh and Vu-Khanh [39] established a phenomenological model based on the Arrhenius function to predict the behavior of polychloroprene rubber during thermo-oxidative aging. Moreover, Johlitz et al. [40] presented a physically-based model to simulate the mechanical response of automobile bearings during thermo-oxidative aging. The aging phenomenon can also be modeled using molecular dynamics simulations, although it requires higher computational costs, implementation of either coarse-graining schemes [41, 42] or multi-scaling approaches [43] could accelerate these simulations. The focus of this work is hygrothermal aging which is the interaction of elastomeric component with moisture in the presence of oxygen and temperature. Hygrothermal aging is a combination of chemical and physical aging that changes the polymer matrix over time. In turn, this change of the polymer matrix can directly affect the mechanical behavior and increase chances of premature failure. In this paper, effects of hygrothermal aging on nonlinear mechanical responses of rubber-like materials, in particular, the idealized Mullins effect and permanent set has been modeled. To the best of the authors' knowledge, currently, there isn't any model that can predict synergies effects of thermo-oxidative aging (i.e. oxygen) and hydrolytic aging (i.e. water) on the constitutive response of polymeric matrix. This paper is outlined as follows, first, the experimental characterization is discussed in section 2. The constitutive model is discussed in section 3, while the validation and results are presented in section 4. Finally, conclusion is presented in section 5.

2 Experimental Characterization

The Ethylene Propylene Diene Monomer (EPDM) rubber in this study was procured as rectangular sheets of nearly 0.0625" thickness from one supplier. Mechanical tests were performed by a uni-axial universal testing machine (TestResources 311 Series Frame). Tests were done in a displacement control manner with a very slow strain rate (i.e. $43.37 \frac{\%}{min}$) to assure the quasi-static condition. The distance between the extensometer grips was set to 25.4mm and all the experiments were performed at room condition (i.e. $22 \pm 2^\circ C$, $50 \pm 3\% RH$). Each test has been repeated with 3 samples for reliability control.

Hydrolytic aging. To simulate the hydrolytic aging condition, samples were fully immersed in sealed containers filled with distilled water ($pH = 7$) under constant pressure. Elevated temperatures of 80° , and $95^\circ C$ were utilized to simulate accelerated

aging conditions.

Hygrothermal aging. To achieve hygrothermal aging, saturated saltwater solutions were utilized to maintain particular values of relative humidities inside sealed containers. Two different humidities of 0%, and 80%, two temperatures 80°C, and 95°C and two aging duration 20, and 30 days were adopted for this aging experiment (see Fig. 1).

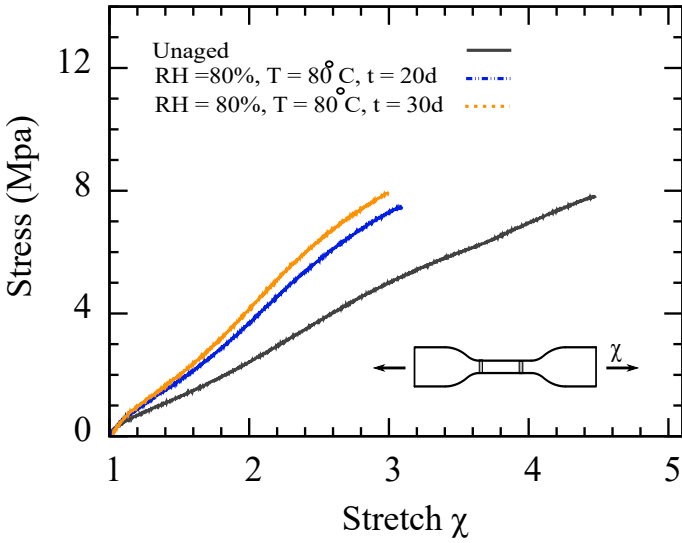


FIGURE 1. The constitutive response of EPDM specimens aged at temperature 80°C, and relative humidity of 80%.

Based on experimental studies on the nature of chemical reactions during aging, chain scission and bond creation happen simultaneously [40]. However, the rates of these two are different. Thus, during aging, cross-link density might increase or decrease depending on the rate of reactions. For instance, during thermo-oxidative aging, the cross-linking is dominant to chain scission which results in amplification of young modulus and reduction of elongation at the break while [44] in hydrolytic aging the process is vice-versa [17]. Accordingly, similar to the thermo-oxidative aging, here the cross-link density is increased which results in the increase of the young's modulus.

3 Constitutive Model

Hygrothermal aging is a kind of environmental damage that results from two simultaneous sub-aging mechanisms, namely, thermo-oxidative and hydrolytic aging. We consider thermo-oxidative aging to be governed by two competing micro-mechanisms [45]: chain scission and cross-link formation, while hydrolytic aging is considered as the result of parallel reduction

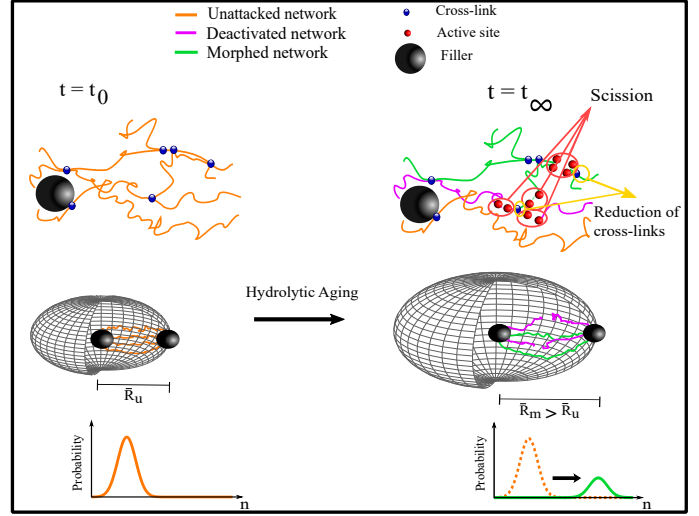


FIGURE 2. A schematic diagram of hydrolytic aging. Hydrolytic aging is due to the reduction of cross-links and chain scission and it can be modeled with a new network with longer chains [26].

in the active chains and cross-links [17] (see Fig. 2). To characterize the effects of aging time on the constitute behavior of polymer matrix, we defined two end-states of the material as the state of polymer matrix at initial state Ψ_0 and fully aged state at time infinity Ψ_∞ . The end-states are considered as extreme points of aging, and accordingly, strain energy of the material in all other states of aging can be calculated through predefined shape functions, $N(t, T)$ and $N'(t, T) = 1 - N(t, T)$, as

$$\Psi_M(t, T, RH, \mathbf{F}) = N(t, T)\Psi_0 + N'(t, T)\Psi_\infty, \quad (1)$$

where $N(t, T)$ is the shape function that represents the contribution of each initial state in the current state. In most solo aging of the polymeric systems, the evolution of the shape function can be well captured by the Arrhenius decay function [46] as

$$N(t, T) = \exp\left(-\gamma \exp\left(-\frac{E_a}{\mathcal{R}T}\right)t\right). \quad (2)$$

Here, γ is the underlying degradation rate, E_a the activation energy, and $\mathcal{R} = 8.314[J/mol][K]$ the universal gas constant. The main challenge is to define the status of the polymer matrix at the fully aged state Ψ_∞ . Here, based on the theory of network decomposition, we assume that fully aged polymer matrix decomposes into two different networks; one of them resulted from the diffusion of oxygen to the polymer matrix Ψ_i^∞ , and the second one modeled the effect of water on the polymer matrix Ψ_h^∞ . Hence, the infinity state of the polymer matrix can be written as

$$\Psi_\infty = (1 - \beta)\Psi_i^\infty + \beta\Psi_h^\infty, \quad (0 \leq \beta \leq 1), \quad (3)$$

where $\beta(t, T, RH)$ defines the portion of each network, which contributes to the complete aging of polymer matrix for a certain amount of aging temperature and humidity. The hydrolysis network Ψ_h^∞ , itself, decomposes into two independent sub-networks namely morphed Ψ_m^∞ and deactivated networks Ψ_d^∞ (see [26] for details) and can be written as

$$\Psi_h^\infty = \alpha \Psi_m^\infty + (1 - \alpha) \Psi_d^\infty, \quad (0 \leq \alpha \leq 1), \quad (4)$$

where the parameter α depicts the contribution of each sub-microstructural phenomenon and is considered as a fitting parameter. The next step is to find the portion of each aging mechanism (i.e. the parameter β), which is involved in the construction of the polymer elasticity. In this respect, new experimental studies reveal that the more water in the polymer matrix, the lower T_g it has and, as a result, the more plasticizing damage. Hence, the parameter β should be a function of water absorption. Within this concept, experimental observations confirm that the water uptake is a nonlinear function of time t , temperature T , and humidity RH and could be best modeled via Arrhenius function. Hence one can define the function $\beta(t, T, RH)$ as

$$\beta(t, T, RH) = \operatorname{erf}\left(\frac{RH}{Q}\right) \sqrt{\theta \exp\left[-\frac{E_b}{RT}\right] t}, \quad (5)$$

where the parameter E_b is the activation energy which is attributed to the water diffusion, and parameters Q and θ are adjusting parameters that keep the range of $\beta(t, T, RH)$ between 0, and 1 (i.e. $0 \leq \beta \leq 1$). The lower bound of the β (i.e. $\beta = 0$) indicates that there is no humidity effect on mechanical response and only thermal degradation Ψ_b causes the matrix deterioration.

3-D generalization Integrating the entropic energy of a sub-network in all direction, the macroscopic energy of an arbitrary three-dimensional matrix can be written as

$$\Psi_\bullet = \frac{1}{A_s} \int_S \psi_\bullet d\mathcal{A} \cong \sum_{i=1}^k \psi_i w_i, \Rightarrow$$

$$\psi_M^d = N(t, T) \psi_0^d + (1 - \beta) N'(t, T) \psi_b^d + \alpha \beta N'(t, T) \psi_m^d, \quad (6)$$

where the parameter A_s is the surface area of unit sphere, \mathcal{A}^d the unit area normal to the direction d , w_i Gaussian weight coefficients according to the direction d_i ($i = 1, 2, \dots, 45$), and ψ_\bullet represents the total energy of each network in direction d .

4 Validation and Results

The model is a kind of modular platform to describe damage accumulation in the course of hydrothermal corrosion. Each network is responsible to model the energy reduction of sub-aging

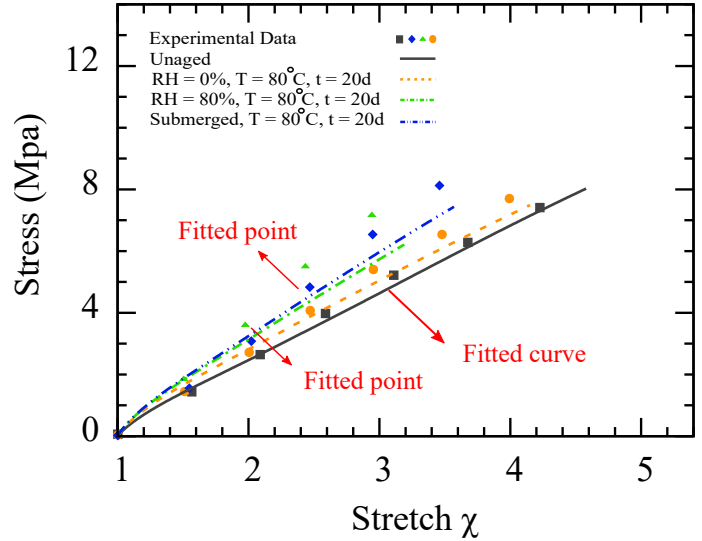


FIGURE 3. Validation of model predictions for EPDM rubber in the unaged state, thermo-oxidative (i.e. $RH = 0\%$), hydrothermal (i.e. $RH = 80\%$), and hydrolytic aging, for 20 days aged samples at temperature 80°C

mechanisms, i.e. i) hydrolytic, and ii) thermo-oxidative. The correlation between these two independent aging mechanisms is held by a nonlinear function of time, temperature, and relative humidity. Therefore, each network should be fitted to the experiments separately. Figs. 3, 4, 5, and 6 shows the prediction of the devised model which were bench-marked against our experimental results, while the Table 1 depicts the so-obtained material parameters.

TABLE 1. Material parameters of the devised model for EPDM rubber

$\mathcal{N}_0 k_b T \bar{R}_0$	\bar{R}_m	\bar{R}_T	μ_0	μ_m	μ_T	σ	
1.927	2.65	3.86	2.34	8.04	8.06	5.63	3.27
v	α	Q	γ	θ	$\frac{E_a}{\mathcal{R}}$	$\frac{E_b}{\mathcal{R}}$	
1.009	0.7639	80	$5.42\text{e}+12$	$3.2\text{e}-1$	$1.17\text{e}+04$	$0.74\text{e}+03$	
			[1/day]	[1/day]	[K]	[K]	

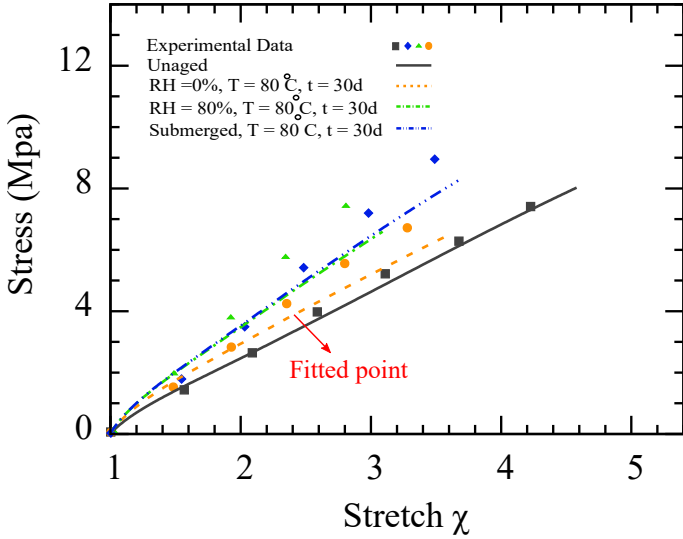


FIGURE 4. Validation of model predictions for EPDM rubber in the unaged state, thermo-oxidative (i.e. $RH = 0\%$), hygrothermal (i.e. $RH = 80\%$), and hydrolytic aging, for 30 days aged samples at temperature 80°C

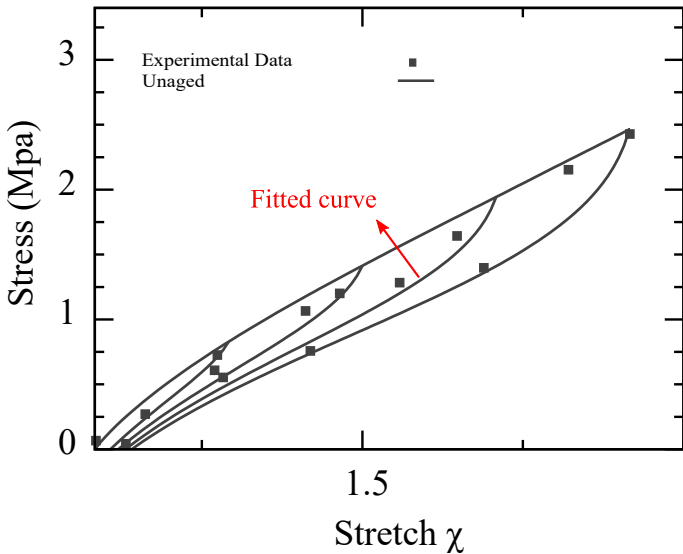


FIGURE 5. constitutive behavior of virgin sample.

5 Conclusion

In this paper, the effect of Hygrothermal aging on a nonlinear mechanical responses of rubber-like materials, in particular, the idealized Mullins effect and permanent set have been modeled. Hygrothermal results from the interaction of elastomeric component with moisture in the presence of oxygen and temperature. The effect of hygrothermal damage on the mechanical integrity of the polymer matrix can be considered as the result

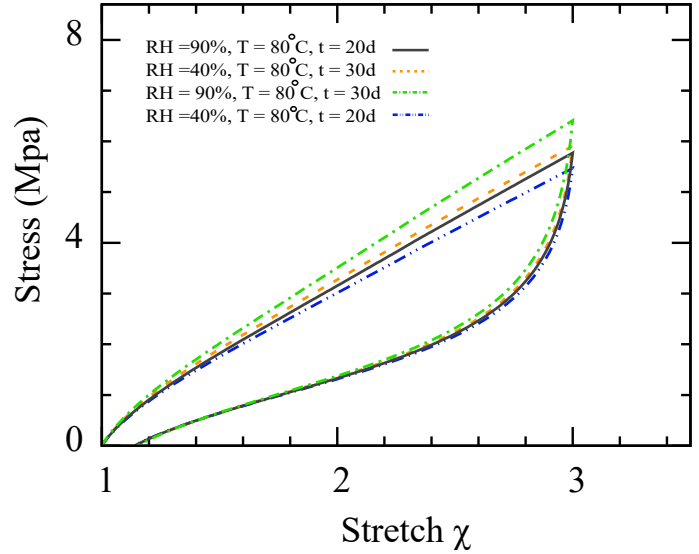


FIGURE 6. Prediction of idealized Mullins effect behavior with respect to storage time t and humidity RH .

of damage accumulation of two independent aging mechanism namely; i) thermo-oxidative, and ii) Hydrolytic aging. In order to capture the mutual effects of thermo-oxidative and hydrolytic aging, the assumption has been made that each of the aging phenomena can be superposed to each other. In fact, each of them works independently and as a result, they can compete with each other. The strain energy function of the polymer matrix is written with respect to four independent mechanisms; i) the shrinking original matrix that has neither been attacked by water nor oxygen, ii) conversion of the first network to two new network due to the reduction and formation the cross-links, and iii) energy loss from network degradation due to water attacks to polymer active agents. Moreover, the proposed model is based on the assumption of homogeneous diffusion and mainly suitable for thin samples. Finally, the model is validated with respect to extensive sets of our experimental data. In view of its interpret-ability, precision, and deep insight it provides into the nature of damage accumulation, the model is a good choice for advance implementation in FE applications.

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