Rubber aging life prediction based on interpolation and improved time-temperature superposition principle

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Abstract
With focus on quickly and accurately predicting and evaluating the aging performance degradation of rubber at room temperature, the pseudo-failure life at each different acceleration temperature is proposed to be calculated by interpolation method based on indoor high temperature accelerated aging data, and on the basis of the obtained pseudo-failure life. By introducing the time-temperature equivalence principle, a shift factor obeying to an Arrhenius law is derived, and master curves are built as well for the compression set as for the ultimate mechanical properties. The concept of the sum of squares of dispersion coefficient errors is proposed to evaluate the prediction accuracy. Meanwhile a quantitative calculation method that considers the effect of temperature on the performance degradation curve and the shift factor is innovatively proposes. The results show that the proposed optimization method based on the traditional time-temperature superposition principle can quickly process the aging life at room temperature, and the prediction results are distributed within the 3-fold dispersion line, which can well meet the engineering requirements. The reduction of the DSC value from 1.4164 to 1.0828 further demonstrates the effectiveness of the proposed method above. This method can provide some reference for other related polymer materials accelerated aging data processing and life prediction.

Nomenclature
(HD) Hexadiene
(NR) Natural Rubber
(IR) Isoprene Rubber
(BR) Butadiene Rubber
(CLD) Cross-Link Density
(WLF) Williams-Landel-Ferry
(SED) Strain Energy Density
(NBR) Nitrile Butadiene Rubber
(SBR) Styrene Butadiene Rubber
(NMR) Nuclear Magnetic Resonance
(EPDM) Ethylene Propylene Diene Monomer
(HNBR) Hydrogenated Nitrile Butadiene Rubber

1. Introduction
Rubber materials are widely used in various industrial fields due to their excellent properties, where rubber parts assume the roles of vibration damping, insulation, sealing and transmission, respectively. In the process of
rubber storage and use, the mechanical properties of rubber components are gradually deteriorated by external environmental factors such as light, temperature, humidity, salt spray, oil and mechanical stress until they lose their value of use. Rubber seals are often in the key part of the product, but also the weak link of the product, the failure of the seals will lead to the entire product can not work properly, or even cause a major safety accident. Therefore, it is of theoretical significance and practical engineering value to carry out environmental aging research on rubber products with different formulations, vulcanization processes and structural dimensions, to explore in depth the accelerated aging test methods and aging life prediction, and to propose new methods for aging protection of rubber products, which is also of great importance to the selection of rubber and the prevention of accidents.

This aging process results mainly from the heat due to hysteric loss, which affects not only the material property but also the service lifetime of rubber [1]. The research on accelerated aging and life prediction methods of rubber needs to be further deepened, correlation analysis of data under different accelerated aging conditions and natural aging, exploration of new life prediction theories and methods can provide theoretical basis and practical reference for performance monitoring, maintenance and replacement of rubber parts [2]. Due to the wide application of rubber materials, a lot of research has been conducted at home and abroad on the aging of materials and components, and the overall can be summarized into four aspects, such as aging micromechanism research, macro mechanical property analysis, accelerated aging life prediction and anti-aging protection method research. The microscopic researches compare the degree of aging of the samples by thermogravimetric analysis, infrared spectroscopy and scanning electron microscopy and also explain the aging mechanism from the perspective of molecular dynamics [3–7]. The effect of ZnO particle size on the crosslinking and thermal aging behavior of natural rubber (NR) was investigated at the microscopic level by Lee et al [3]. The results showed that the ZnO-filled nanoparticles had higher crosslinking density, lower polysulfide crosslinking, and stronger mechanical properties compared with the micron ZnO-filled NR vulcanize filler material. After thermal aging, the NR filled with nanoparticle ZnO exhibited more stable chemical and mechanical properties. Eunjoo Kong et al [4] partially replaced the carbon black filler in natural rubber with graphene, and the hardness and compression properties increased linearly with the amount of CB partially replaced by graphene (1–5 phr: per hundred rubber parts). Starting from the relationship between thermal oxygen temperature and lifetime, Arrhenius plots were drawn by the least squares method to predict the lifetime of NR composites, and the conclusion that the activation energy of NR composites reached a maximum when filled with 3 phr graphene was obtained, which provided some theoretical reference for the improvement of fillers. Sima Kashi et al [5] conducted accelerated degradation tests at high temperatures (195 °C) on silicone rubber exposed to a diol environment and showed that both time and temperature had a significant effect on the mechanical properties, with tear strength and hardness showing an initial increase in the early stages of aging, followed by a decreasing trend. In contrast, thermal properties did not change significantly, and FTIR did not detect any changes in rubber surface chemistry with aging. However, SEM provided evidence of increased brittle behavior of the fracture surface morphology. The study method in literature [6] is similar to that of literature [4], the difference lies in the difference of the study object and the filler, which changed to hydrogenated nitrile rubber nanocomposites and the filler is organo-modified clay. Using nitrile carbon black-filled rubber as the object of study, Elena V. Bystritskaya et al [7] used and verified the feasibility of the thermogravimetric analysis method that can be used to predict the thermal stability of rubber. The macroscopic study of rubber materials or components mainly analyzes the macroscopic mechanical properties of rubber samples, which mainly includes the changes of properties such as constitutive relationship, breaking stress, elongation at break, compression set, elastic modulus and hardness under different aging temperatures and different aging times [8–16]. In the macroscopic study, WOO et al [8] studied EPDM rubber parts and NBR rubber parts in refrigerators and explored the effect of thermal and oxygen aging on the performance of rubber materials, and proposed a prediction method for effective life based on the Arrhenius equation and experimental results. N. Rodriguez et al [9] conducted an experimental study of unaged and aged specimens of butylene-isoprene rubber compounds (SBR-IR) and found that aging had a small effect on their friction and adhesion properties, but showed a significant decrease in elongation at break and toughness with aging, attributed to changes in crosslink density, reduction in strength of the filler matrix, and crack formation and growth. P. Pourmand et al [10] investigated the effect of thermal-oxidative aging on filled EPDM materials and proposed the use of NMR spectroscopy for analysis to assess the gradient of structural and property changes with aging, and the validity of this monitoring method was further demonstrated by the correlation of data obtained from NMR and indentation modulus analysis with fracture strain data. Christoph Neuhaus et al [11] investigated the indirect and direct effects of high temperature aging effects on fatigue life and proposed a new model that takes these effects into account, which helps to establish more realistic testing procedures for the development of rubber components and provides more realistic information for improving fatigue life methods. Byungwoo Moon, et al [12] obtained the crosslink density by performing swelling tests on aged NR/BR blended rubber compounds based on the test results jointly using the Flory-Rehner equation, and there is a linear relationship between strain energy density (SED)
and cross-link density (CLD) when the increase in cross-link structure is represented by a parallel spring model. Finally, a method for predicting the aging behavior of NR/BR blends using cross-link density is proposed using the relationship between strain energy density and cross-link density as an equation. T. Ha-Anh et al. [13] found that the dependence of mechanical behavior on aging time and aging temperature followed an Arrhenius relationship with similar activation energy by studying the effect of thermal-oxidative aging on the mechanical properties of neoprene. Hüsnü Dal et al. [14] proposed micro-continuum material model which is based on a serial construction of a Langevin-type spring representing the energy storage owing to conformational changes induced by deformation, to a bond potential representing the energy stored in the polymer chain due to the interatomic displacement. For the representation of the micro-macro transition, the non-affine kinematics of the micro-sphere model is used. A novel numerical scheme for the FE implementation of the proposed model is demonstrated. M. Ben Hassine et al. [15] proposed a method to predict the tensile ratio of rubber parts at thermal aging failure by introducing the principle of time-temperature superposition, deriving a shift factor obeying Arrhenius law, and constructing a master curve for the mean molar mass and mechanical properties, and determining the relationship between the fracture energy and the square root of the mean molar mass by fracture mechanics tests. BenAlcock et al. [16] investigated the effects of aging in oil media on the tensile properties, apparent crosslink density, carbon dioxide diffusion, and Shore hardness of typical carbon black reinforced hydrogenated nitrile butadiene rubber (HNBR). The results show that tensile stiffness and hardness increase with increasing apparent crosslink density, while carbon dioxide diffusion and saturation swelling in chloroform decrease. Geometry-independent tests (e.g., solvent swelling) can be performed on parts recovered from use to predict properties (e.g., tensile stiffness or gas diffusion) that are not typically measured on recovered parts due to geometric limitations. According to the existing accelerated aging test research results, it is found that the oven accelerated aging test is the closest to natural aging, and the accelerated aging test is often used to obtain aging data. Rubber accelerated aging research primarily aims to make predictions about the ageing life of rubber, thus providing reference for rubber reliability assessment. Different approaches are used to establish the evaluation model of aging life to achieve the purpose of predicting rubber products, and common modeling methods include WLF equation [17], Arrhenius method [18] and ternary model [19]. Compression set as one of the often used aging performance degradation indicators has the advantages of easy measurement, monotonic data, and easy modeling of degradation trajectories, and dynamic modeling is often used to characterize the relationship between performance degradation and aging rate [20–22]. The prediction of fatigue life of rubber aging has also been studied by domestic and foreign scholars. Cho et al. [23] used a combination of nonlinear finite element and ε – N methods to predict the fatigue life of EPDM, Lay et al. [24] studied the aging life of EPDM seals in complex environments by accelerated aging tests and Arrhenius method, and Koemmling et al. [25] used time and temperature conversion and Arrhenius method to predict the life of O-ring seals.

The time-temperature superposition principle has not been used in conjunction with interpolation methods for life prediction in existing microscopic or macroscopic studies of rubber materials or components [3–16] and these studies are mainly based on linear Arrhenius approach [8, 9, 13, 15], which rarely involved in the non-Arrhenius behavior. In this paper, based on the accelerated thermal and oxygen aging and natural aging data of 8106 ethylene propylene rubber as the test data, the compression permanent deformation was used as the aging performance evaluation index, the mathematical model of rubber aging was established by applying the time-temperature superposition principle, and the natural aging life at room temperature was predicted by combining this mathematical model with the high-temperature aging data, and the existence of a certain degree of non-Arrhenius behavior was obtained based on the accelerated aging test data. Arrhenius behavior was obtained based on the accelerated aging test data, and the model was further optimized by introducing the characterization of non-Arrhenius behavior on the basis of the life prediction model at room temperature. The concept of the sum of squares of dispersion coefficient errors is proposed to evaluate the prediction accuracy. According to the comparison with the measured natural aging life, the improved model prediction is significantly better than the traditional Arrhenius equation based on the time-temperature superposition principle method.

2. Experimental

2.1. Determination of samples and aging degradation performance indicators

Ethylene propylene diene monomer (EPDM) rubber studied in the work was a commercial elastomeric material, which contained 1, 4-hexadiene (HD) as diene. The EPDM was composed of 51.6 wt% ethylene, 39.9 wt% propylene and 7.7 wt% HD, and it was mainly used as the seals of using in the design of launch vehicle structures and power systems and automobile parts. According to the working mechanism of sealing materials, the monotonic change of compression set, which is directly related to the sealing performance, makes it more suitable as an indicator of the degree of rubber aging. In accordance with GB/T 7759-1996 processing of rubber...
compression deformation specimens and the corresponding test tooling, the specimens are subjected to the same stress conditions as the actual [26]. Compression permanent deformation specimens are made in accordance with GB/T 7759.1-2015 for type A specimens, and the dimensional drawing of rubber specimens for testing is shown in figure 1. Figure 2 is the physical diagram of the accelerated aging test oven, at least three specimens are used at each accelerated temperature to carry out the test, and the average value of the compressive permanent deformation is taken as the final evaluation index of the aging performance. The fixture used for the test is shown in figure 3.

2.2. Natural aging and accelerated aging test methods
The specified compressive stress is applied to the rubber specimen before the test starts, and after 24 h at room temperature, the fixture is released so that the specimen is placed in the free state for 1 h to measure its recovery height before aging, and the compression is continued to the specified deformation rate, waiting for the aging test to be carried out. Natural aging test will be the above compression tooling stored in 10–25 °C, relative humidity of less than 75%, no direct sunlight, no acid, alkali or other corrosive environment. After the test period, remove the test fixture, release the fixture and let the specimen stand in the free state for 1 h, then measure the return height after compression and calculate the compression set. Accelerated aging test is carried out in accordance with GJB92.1-1986. The accelerated aging test selects temperature as the accelerating stress. The accelerated stress temperature is set to 80–110 °C, and every 10 °C interval is used as an accelerated stress. Accelerated aging test scheme is designed as shown in table 1. The T column in table 1 indicates the temperature constant stress and accelerated temperature stress settings, and the Point1-Point8 settings correspond to the time points of rubber aging performance testing under different stresses, respectively. To set the test cycle from the aging chamber to take out the tooling, cooling at (25 ± 1) °C for 2 h, then loosen the fixture, so that the specimen in a free state for 1 h, measuring its return height after compression, and calculate the compression set.
2.3 Data processing in accelerated test
For the sealing rubber under study, the compression set is the most appropriate measure of its aging degree because it is in compression during normal operation. The measurement of compression set is based on the provisions of ‘Determination of compression set of vulcanized rubber or thermoplastic rubber’ (GB/T7759-2015). The expression of compression set rate is shown in equation (1)

\[ \varepsilon = \frac{h_0 - h_1}{h_0 - h_r} \times 100\% \]  

\(h_0\) is the height of the unaged sample, \(h_1\) is the height of the limiter, \(h_r\) is the height of the aged sample.

3. Results and discussion
The requirements for the test samples are produced and processed in accordance with the rubber product process, and the physical and chemical properties are retested, and the rubber material samples for accelerated aging and natural storage aging are the same batch of products, and the uniformity of sample batches, test equipment and test personnel ensures the validity of the test results.

3.1 Accelerated aging and natural aging test results
The test data of the compression set of the material after the natural aging test and the thermal oxygen accelerated aging test for 8106 ethylene propylene rubber are shown in table 2.

3.2 Modeling of rubber aging life based on the conventional time-temperature superposition principle
Choose 80 °C as the reference temperature, that is, the shift factor corresponding to 80 °C is defined as 1. Take the critical performance parameter change of aging as the compression permanent deformation reaches 60%, calculate the critical aging time under 4 groups of accelerated temperature. Based on the measured high-temperature accelerated aging data, the aging time at each different temperature when the performance deteriorates to a critical value is calculated by interpolation, and the shift factor at different temperatures can be obtained by calculating the ratio of the reference temperature to the aging time at each aging temperature. Figure 4 shows the shift factor at each temperature. According to the data distribution of the shift factor in

![Test fixture.](image-url)
After shifting the high-temperature accelerated aging data to 80 °C, it can be seen that the linearity of the panned data is high, which indicates that the rubber aging mechanism at each accelerated temperature does not change, and this phenomenon also indicates the feasibility of using the time-temperature superposition principle for rubber aging life prediction. The extrapolation of the shift factor at high temperature to obtain the translation factor at room temperature is shown in Figure 5. The acquisition of this shift factor considers the finger front factor in the Arrhenius equation as well as the activation energy to be constant in the range of predicted and tested temperatures, i.e., independent of temperature, while a deeper study finds that the finger front factor in the Arrhenius equation and the activation energy in the Arrhenius equation are temperature dependent, and a correction for this deficiency is proposed in section 3.3.

The life prediction curve at room temperature is based on the time-temperature superposition principle, and the life prediction curve at room temperature is firstly shifted to the reference temperature for all the curves at the accelerated temperature, and then the comprehensive performance curve is obtained by regressing all the curves shifted to the reference temperature and further extrapolated to room temperature, which makes full use of all the data and can minimize the average error of the extrapolated data. The prediction results at room temperature are measured by the sum of squared dispersion coefficient errors, which is defined as shown in equation (2), where \( pd \) is the natural aging life prediction data, \( md \) is the measured life data, and \( DCS \) is the sum of squared dispersion coefficient errors. \( i \) takes values from 1 to 6 respectively, representing 6 time measurement points. Figure 6 shows the predicted aging life by the time-temperature superposition principle compared with the measured life, and the predicted life distribution is mostly within the 3-fold dispersion line. The reasons for the large errors can be attributed to two points: the assumption of temperature-independent constants for the activation energy in the proposed prediction method and the measurement errors. The effect of measurement error is not considered in this study. Figure 7 shows the aging data at each accelerated temperature and the aging curves at room temperature derived from the time-temperature superposition principle. As can be seen from figure 7, the aging curves at each accelerated temperature are straight lines in logarithmic coordinates, and the slopes are basically the same, with different intercepts. The translation factor calculated from the time-temperature superposition principle can determine the linear intercept at room temperature, and thus obtain the aging life prediction equation at room temperature. The comparison of the measured data at each temperature in figure 7 further demonstrates that the activation energy varies slightly at different reaction temperatures.

![Figure 4](image-url)  
**Figure 4.** Fitting of aging performance change at high temperature advection to reference temperature.

**Table 2.** Compression set at different temperatures.

| T(°C) | Point1/d | Point2/d | Point3/d | Point4/d | Point5/d | Point6/d | Point7/d | Point8/d |
|-------|----------|----------|----------|----------|----------|----------|----------|----------|
| 25    | 0.0364   | 0.2411   | 0.2894   | 0.3322   | 0.5147   | 0.6644   | —        | —        |
| 80    | 0.1800   | 0.2430   | 0.2960   | 0.4010   | 0.4850   | 0.5310   | 0.5750   | 0.6280   |
| 90    | 0.2440   | 0.3350   | 0.4190   | 0.5070   | 0.5800   | 0.6500   | 0.6910   | 0.7730   |
| 100   | 0.3090   | 0.4370   | 0.5290   | 0.5930   | 0.6390   | 0.7330   | 0.7900   | 0.8430   |
| 110   | 0.2920   | 0.4930   | 0.5860   | 0.6940   | 0.7410   | 0.8010   | 0.8620   | -        |
Figure 5. High temperature advection factor and extrapolation.

Figure 6. Natural aging prediction and measured data distribution.

Figure 7. Comparison of measured aging data by temperature.

\[
DCS = \sum \left( \frac{pd_i}{md_i} - 1 \right)^2
\]
3.3. Improved modeling and validation of rubber aging life

Based on the life prediction method proposed above based on the time-temperature superposition principle, the effect of temperature on the activation energy is taken into account in the prediction model, i.e., it has a certain effect on the slope of the performance degradation curve and the extrapolation to the translation factor at room temperature. The calculation of the slope of the degradation curve at room temperature and the translation factor considering the temperature effect is shown in equation (3) and equation (4). In equation (3), $M_k$ denotes the slope of the integrated curve of performance degradation at room temperature after optimization, $k$ is the slope of the integrated curve at room temperature obtained by the original prediction method, $MAT$ in equation (4) is the flattening factor at room temperature obtained after optimization, $AT$ is the flattening factor at room temperature before optimization, $\alpha$ and $\beta$ are coefficients introduced to characterize the degree of influence on the slope of degradation and flattening factor when considering temperature changes, respectively. $T_1$ is the temperature at room temperature and $T_0$ is the reference temperature.

\[
M_k = k \left( \frac{T_1}{T_0} \right)^\alpha \tag{3}
\]

\[
MAT = AT \left( \frac{T_1}{T_0} \right)^\beta \tag{4}
\]

The distribution of the optimized natural aging life prediction and the measured data is shown in figure 8.

The specific life prediction data with dispersion factors are presented in table 3. In table 3, $md$, $pd1$, $pd2$, $DCS1$, $DCS2$ indicate the measured data at room temperature, the life prediction data before and after optimization, and the life prediction dispersion coefficient before and after optimization, respectively.

The error sum of squares of dispersion coefficients before optimization is 1.4164, and the error sum of squares of dispersion coefficients after optimization is 1.0828. And the difference of dispersion coefficients is also reduced, and this phenomenon also proves that the proposed optimization method has a significant improvement effect on the overall prediction accuracy.

Table 3. Comparison table of prediction accuracy before and after optimization.

| $md$  | $pd1$ | $pd2$ | $DCS1$ | $DCS2$ |
|-------|-------|-------|--------|--------|
| 71    | 64    | 93    | 0.0099 | 0.0930 |
| 926   | 358   | 430   | 0.3761 | 0.2867 |
| 1143  | 538   | 618   | 0.2805 | 0.2110 |
| 2486  | 770   | 852   | 0.4760 | 0.4321 |
| 3491  | 3581  | 3349  | 0.0007 | 0.0017 |
| 8289  | 12 623| 10 291| 0.2733 | 0.0584 |

Figure 8. Distribution of natural aging prediction and measured data after optimization.
4. Conclusion

Based on the interpolation and time-temperature superposition principle, the rubber aging shift factor at room temperature was obtained by extrapolation from the high temperature accelerated aging data, and the storage life evaluation equation of rubber at room temperature was firstly established assuming that the activation energy does not change with temperature. On this basis, the improved life prediction equation was established by considering the influence of temperature on the slope and translation factor of the degradation curve. The prediction accuracy and the overall effect of prediction are obviously due to the results obtained based on the traditional time-temperature superposition principle. The effectiveness of the proposed model was verified by experiments. The main conclusions of this research include the following:

- The comparison of accelerated aging test and natural aging test results found their basic agreement, and the deviations of the optimized evaluated life were kept within 3 times the dispersion line, which has high application reference value.
- The reasons for the poor prediction accuracy and overall prediction before optimization are mainly due to the assumption of constant activation energy at different reaction temperatures and measurement errors.

The next research can introduce multi-factor accelerated aging tests, consider the statistical distribution characteristics of aging life at different temperatures, reasonably select aging evaluation indexes, and carry out quantitative research between macroscopic performance and microscopic mechanisms. Meanwhile, in order to further improve the accuracy of the life prediction methods of rubber sealing materials, accelerated aging test databases and natural aging performance test databases can be established for different types of rubber sealing materials to provide correction amounts for the evaluation results of accelerated aging tests.

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Data availability statement

No new data were created or analysed in this study.

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