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Low-Temperature Performance Testing and Application Optimization of Rubber Product

Jingfu Zhou, Fengyun Yuan, Xiaodong Li, Weidong Liu, Lijian Xu

Qingdao Product Quality Testing Research Institute, Rubber and Tire Department, Qingdao, Shandong, 266100, China

ABSTRACT
This study focuses on rubber products' performance degradation mechanism and optimization strategy in low-temperature environments. Aiming at the key failure problems such as low-temperature embrittlement and dynamic property attenuation, a three-level testing technology system is established by multi-scale mechanism analysis. Based on the failure mechanism, the material optimization path was proposed: the low-temperature toughness was improved by the design of molecular chain flexibility, the directional reinforcement system of nanofillers was constructed to inhibit the propagation of microcracks, the curing process was optimized to control the uniformity of cross-linked network, it is expected to provide theoretical basis and technical support for the reliability improvement of rubber product in extreme environment, and promote the design and standardization process of low-temperature adaptive materials.

KEYWORDS
Rubber product; Low-temperature environment; Performance testing

1 Introduction

Rubber materials are widely used as a key engineering component in aerospace, polar equipment, and new energy fields. In low-temperature service environments, the limited movement of molecular chain segments leads to a sharp rise in the embrittlement of materials, which leads to safety hazards such as sealing failure and dynamic fatigue. Existing studies have shown that when the temperature is close to the vitrification transition point, the free volume shrinkage of the polymers triggers the chain segment freezing, and plasticizer phase separation further accelerates the performance degradation. However, the traditional testing methods make it difficult to simulate the coupling conditions of extreme temperature change rate and multiaxial stress, resulting in the lack of an accurate target for material optimization. There are double limitations in the current research: the quantitative relationship between microscopic structure evolution and macroscopic properties has not been established, especially since the debonding mechanisms of filler-matrix interface at low temperatures are not clear; at the same time, material modification is more dependent on empirical formulations, there is a lack of molecular scale oriented design guidance. With the development of frontier fields such as deep space exploration and liquid hydrogen storage and transportation, rubber materials with low-temperature toughness, fatigue resistance, and environmental adaptability have become a bottleneck restricting technological breakthroughs[1]. Therefore, it is urgent to systematically analyze rubber materials' cross-scale low-temperature failure mechanism, establish a working condition suitability testing system, and then guide the gene-level optimization design of materials.

2 Analysis of low-temperature failure mechanism

2.1 The molecule scale

The performance degeneration of rubber materials at low temperatures is due to the progressive inhibition of the molecular chain movement of polymers. When the temperature approaches the glass transition temperature (Tg), the coordinated rearrangement of the molecular chain segment tends to be stagnant, and the rotation potential barrier within the molecular chain segment increases significantly, resulting in a sharp decrease in conformational entropy. In this process, the free volume shrinks nonlinearly with the decrease in temperature, and the intermolecular forces between the molecular chains are enhanced so that the originally slipable chain segment is"Frozen "in the energy potential well. Notably, polar rubber molecular chains(such as the cyanide group in nitrile butadiene rubber)are more prone to form locally ordered regions at low temperatures due to their strong dipole interactions, further impeding chain segment relaxation [2]. On the other hand, when the cross-linked network shrinks at low temperatures, the internal stress concentration occurs, the relative displacement between the network nodes is forced to be locked, and the macroscopic performance is that the material loses the ability of elastic recovery. This molecular-level thermodynamic-kinetic coupling constitutes the fundamental physical mechanism of low-temperature hardening of rubber.

2.2 Microscopic structure

The chain segment freezing at the molecule scale will induce the cascade evolution of the microscopic phase structure. The filler-polymers interface in the rubber matrix is a key carrier for stress transfer. There are critical challenges in the process of low-temperature shrinkage: the difference in the coefficient of thermal expansion between filler particles (such as carbon black silica) and the rubber matrix leads to the differential contraction of the interface, which causes the interfacial debonding micro-zone; at the same time, due to miscibility mutation, plasticizer and other small molecular additives migrate from the matrix to the defect area, forming a weak interfacial layer. More noteworthy is the phase separation behavior of the multiphase system. In the SBR (polymerized styrene butadiene rubber)/NR (natural rubber) blend system, the difference in the glass transition temperature of different components promotes the redistribution of the microscopic stress field, and cracks preferentially initiate and propagate along the phase interface[3]. The damage evolution at the microscopic level presents self-similar characteristics: the initial micro-voids are gradually connected under the action of alternation thermal stress, and finally, a micro-crack network is formed along the boundary of the filler aggregate or crystallization zone, and the micro-crack network is formed, provides a path basis for macroscopic brittle rupture.

2.3 Macro performance

The accumulation of microscopic defects inevitably manifests as a systematic degeneration of macroscopic performance. In terms of dynamic mechanical property, the sharp increase of Young's modulus below Tg reflects the embrittlement of materials, and the peak shift of the loss factor reveals the transition of molecular relaxation modes; in terms of static mechanical properties, the synchronous deterioration of compression set and tensile strength reflects the failure of energy dissipation mechanisms. The attenuation of functional reliability is particularly prominent: under low-temperature compression conditions, the contact stress attenuation of the seal is caused by the sudden decrease of the stress relaxation rate, which leads to the medium penetration channel; the damping elements are out of tune due to the increase of dynamic modulus, loss of vibration damping function. In essence, macro-failure is the inevitable result of micro-damage under boundary condition constraints: geometric constraints (such as seal groove structures) lead to deterioration of the three-dimensional stress state, the penetration of ambient medium (oil/gas) accelerates the chemical degradation of the crack tip. This multi-element coupling failure mode profoundly explains the complex degradation mechanism of rubber products in low-temperature service.

3 Testing technology system

3.1 Main testing layer

The primary testing layer establishes a low-temperature performance benchmark by revealing the material's intrinsic response. The core focus is on dynamic mechanical response spectrum analysis, using wide-ranging temperature dynamic mechanical analysis (DMA) to capture Young's modulus transition and loss factor peak displacement near Tg and quantitatively characterize the chain segment freezing critical point. The dielectric relaxation spectrum (DRS) was used to monitor the dipole orientation relaxation of the samples under an alternation electric field, and the contribution mechanism of secondary transitions to low-temperature toughness was analyzed. At the same time, the low-temperature tensile-recovery cycle test was used to analyze the entropic elasticity degradation inflection point based on the elastic recovery energy integral curve. Such methods constitute the basic performance database, and their technical advantage is to strip the environmental interference, directly correlate the constitutive relationship between molecular motion and macro modulus evolution, and provide the first evidence for the failure mechanism. The main detection layer should pay special attention to the co-design of the testing parameters: the time and temperature equivalence can be decoupled by orthogonal matching of the frequency spectrum and temperature drop rate, while the hierarchical loading of strain amplitude reveals the damage-sensitive threshold in the nonlinear interval.

3.2 The auxiliary verification layer

The auxiliary verification layer deconstructs the generation and evolution path of microscopic defects. The micro-area morphology of the in-situ tensile fracture surface was analyzed by cryo-scanning electron microscope (Cryo-SEM), and the fracture mode transition temperature was inversely deduced according to the Dimple/cleavage characteristic ratio; Micro-focus X-ray computed tomography (μ-CT) enables non-destructive three-dimensional reconstruction, accurately locates the spatial topology of the interfacial debonding zone and the internal microcracks, combined with a digital volume correlation algorithm to quantify the inhomogeneity of the thermal shrinkage strain field. The key breakthrough in this hierarchy lies in the fusion of"Dynamic capture "and"Static traceability": locking the instantaneous microscopic state by liquid nitrogen quenching and achieving the"Dynamic capture "and"Static traceability "by liquid nitrogen quenching, atomic force microscope (AFM) phase imaging techniques were used to map the nano-scale filler-matrix stress transfer efficiency. This technology interprets the macroscopic parameter anomaly of the main testing layer from the spatial dimension and forms a visual verification closed loop of the microscopic deformation mechanism.

3.3 The extreme simulation layer

The extreme simulation layer aims to reconstruct the extreme coupling conditions of the service scenario. A multi-field coupling accelerated test platform was developed, integrating a temperature control module (-196℃ to 300℃), a medium circulation system (hydraulic oil/liquid oxygen), a mechanical dynamic loading unit (axial pulsation + lateral shear), and the performance evolution tracking of the seal in the transient temperature gradient——pressure pulse——chemical erosion synergistic time-varying field is realized. The core technology lies in the failure critical point prediction model. The phase boundary of material migration from viscoelastic state to glassy state is calibrated in the temperature-stress phase diagram, and the distortion law of the three-dimensional stress field inside the component is inversed by finite element analysis (FEA). The scientific nature of this layer is reflected in the timing control of environmental factors: the degeneration path of actual service is accurately reproduced by programming to reproduce the temperature and pressure drastic change load during the day-night temperature difference cycle in cold regions or rocket launch, thus crossing the cognitive gap between single-field tests in the laboratory and complex engineering conditions[4].

4 Material optimization path

4.1 Design of molecular structure

The core contradiction of low-temperature performance of rubber materials lies in the balance regulation of molecular chain segment motional degrees of freedom and cohesive energy. By precisely designing the chemical architecture of the main chain and embedding dynamic flexible units to retain the necessary rigidity, the relaxation behavior of the chain segment at low temperatures can be significantly improved. The siloxane chain segment is an ideal choice for low-temperature toughening due to its unique characteristics of low potential energy barrier of Si-O bond rotation and high variability of bond angle. In contrast, the long alkyl side chains weaken the intermolecular force through steric effects, reducing the energy mutation in the vitrification process. For special-purposed synthetic rubber, using sequence-controlled block copolymerization techniques is critical——the rigid aromatic chain segment provides the physical cross-linking strength. In contrast, the aliphatic flexible chain provides low-temperature ductility, and the two are separated by microphase separation to form a"Rigid and flexible "topology. Chemical modification of side chains should avoid the tendency of low-temperature crystallization of high-polar groups (such as cyanide and halogen) and instead introduce moderately polar ether bonds or ester groups to reduce the intermolecular force while maintaining matrix cohesion. Molecular weight distribution control is another key dimension: narrow distribution polymers can avoid stress concentration caused by preferential freezing of low-molecular-weight components at low temperatures, while ultra-high-molecular-weight-molecular chains inhibit crack propagation through entanglement networks.

4.2 Innovation of compound system

The multi-level interface engineering of composites is the core path to breaking through the bottleneck of low-temperature performance. The surface functionalization of nanofillers needs to meet two goals: one is to construct a strong and tough covalent bond network at the filler-matrix interface through chemical grafting, and the other is to optimize the stress transfer efficiency by adjusting the geometric characteristics of filler（such as diameter-to-thickness ratio and radius of curvature）. After organic modification, the active sites at the edge of the layer silicate can form ion-dipole inter-action with rubber molecules and maintain the interfacial bonding strength at -50℃. One-dimensional carbon nanotubes form a"Molecular steel bar "effect through axial orientation, which hinders the cross-scale propagation of low-temperature microcracks.

4.3 Process control

The manufacturing process determines the quality and stability of the microscopic structure. In the curing process, the step-by-step temperature control strategy was adopted: at the initial low-temperature stage, the scorch time was extended to promote the dispersion uniformity of filler, and at the later high-temperature stage, the gradient distribution of cross-linking density was precisely controlled, the network embrittlement caused by local over-curing was avoided. In the forming stage, the low-temperature pre-stretching technology is introduced, and the directional stress field is applied above the glass transition temperature to induce the orientation and arrangement of molecular chains to improve the low-temperature deformation coordination ability. The post-treatment process releases the residual interior stress by cryogenic cycling annealing, promotes the filler-matrix interface relaxation, and eliminates the processing freezing defect. The process should match the material's relaxation characteristics and the equipment's temperature control accuracy to ensure consistency between the ordering of the microscopic structure and the macroscopic properties.

5 Application scenario adaptation optimization

5.1 Extreme environment seals

In the field of ultra-cryogenic medium sealing (such as liquid hydrogen/liquid oxygen storage and transportation systems), the core contradiction focuses on the sudden change effect of phase change permeability. When the temperature passes through the Tg critical region, the nonlinear collapse of the free volume of the rubber molecular chain leads to the topology reconstruction of the gas permeation channel and the diffusion flux of small molecules (helium/hydrogen) along the interstices of frozen molecular chains jumps exponentially. The traditional homogeneous rubber compound faces the molecule scale mismatch: the strong polarity of nitrile butadiene rubber induces a negative correlation between the molecular chain breaking energy and the coefficient (s) of friction under high-cold dry friction duty, the low-temperature crystallization tendency of fluoro rubber accelerates the stress cracking of sealing edge. The optimization path needs to anchor the gradient design of the composite system. The outer layer uses fluoro silicone elastomer to construct low-temperature leaky barriers, and the intermediate layer introduces graphene-modified butyl rubber to block heat convection; the inner layer uses polyurethane elastomer to maintain the flexible compensation of contact stress.

5.2 Low-temperature dynamic components

The failure of dynamic components such as solar sails hinge of spacecraft and suspended systems of polar vehicles is caused by the coupling mechanism of stick-slip vibration and brittle fracture. In the temperature range of -80°C~-40°C, the dynamic stiffness of rubber-damping elements overshoots due to the sharp increase of Young's modulus, and the vibration energy is transmitted inversely with the metal connector. At the same time, the boundary lubrication phase transition of the interface condensed water film (liquid → amorphous solid) induces the fretting wear caused by the Stick-Slip Effect. The fundamental optimization needs to reconstruct the hysteresis characteristics of the material: the molecular chain flexibility block grafting is used to reduce the loss factor temperature gradient, and the fatigue crack growth threshold is improved by combining the directional reinforcement network of carbon nanotubes. The more critical dynamic adaptation design is to establish the phase matching between the time-varying temperature field and the stress field and dynamically adjust the cross-linking density distribution gradient according to the temperature drop curve of the absorber. The product maintains the stability of damping characteristics under the Arctic Circle's day and night cycle conditions. It suppresses the energy aggregation phenomenon of the vibration spectrum in the characteristic frequency region.

5.3 Energy equipment applications

The lithium cell cryogenic sealing and wind electric pitch system in new energy equipment are facing the challenge of multi-physical field timing deterioration. When the power battery of an electric vehicle is fast charged in cold regions, the expansion force of the battery core and the low-temperature contraction force of the rubber pad produce a vector conflict (axial compression/radially stretching compound stress) at the sealing interface, the "Onion effect "of interface peeling between filler and matrix is induced. The prediction model of the time-dependent strain field should be established: the hyper-elastic constitutive equation should be used to inverse the time-temperature equivalent relation of the curing system to compression permanent deformation; a multi-stage curing process was designed to make the cross-linked network maintain the buffering ability of conformational entropy in the deep and low-temperature regions. For the wind turbine pitch-bearing seal, it is necessary to break through the ice-solid coupling failure mechanism under extreme wind speed. The non-equilibrium centrifugal load generated by blade ice coating causes micron-level oscillating wear on the seal lip. The diamond-like carbon (DLC) coating deposited by magnetron sputtering was used to control the surface desorption potential energy barrier and eliminate the homogeneous nucleation basis of ice crystals on the rubber surface.

6 Conclusion

This study established a multi-scale diagnosis paradigm for low-temperature failure of rubber materials by constructing a progressive testing technology system. The critical laws of molecular relaxation revealed by the main testing layer, the defect evolution topological paths captured by the auxiliary verification layer, and the time-varying multi-field coupling degradation model reconstructed by the extreme simulation layer jointly form a closed loop of failure mechanism cognition from the nano-scale to the macro-scale. In the application dimension, the gradient composite design of the extreme seal blocks the penetration of the phase transition, the hysteresis characteristic spectrum of the dynamic component is modulated to suppress stick-slip chatter, the time-varying stress field matching model of energy equipment eliminates sequential load conflicts.

About the Author

Jingfu Zhou, master, engineer, research direction: Tire and rubber product testing and research.

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