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Progress in Experimental Research on Mechanical Behavior of Composite Solid Propellants

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Abstract: Composite solid propellant is a particle reinforced composite material, and its microstructure strongly affects its macroscopic mechanical properties. This article reviews the current research status of macroscopic mechanical experiments and microscopic experiments. In terms of macroscopic mechanical performance testing, based on the description of the number of loading axes used to load the specimen, the development of related experimental research on uniaxial loading is relatively mature, while research on multi-axial loading is still lacking, and a unified standard has not yet been formed. In terms of microscopic experiments, although there are currently many observation methods, each has its own advantages and disadvantages; In experimental studies on the microstructure observation of solid propellants under loading conditions, most of them are based on uniaxial loading devices combined with optical microscopes, scanning electron microscopes, or micro CT devices to conduct research on the microstructure damage process of solid propellants under uniaxial loading. However, the research on the microstructure damage evolution process of solid propellants under multi-axial loading is currently in a research gap at home and abroad. Finally, this article summarizes and comments on the current research status, Proposed the shortcomings of current research and the key areas that need to be studied.

Keywords: Solid propellant, Macro and micro scale, Mechanical tests, Observation methods, Multi axis loading.

1. Introduction

The phenomenon of changes in the relevant performance of the research object caused by size is usually referred to as the size effect. This can be divided into three levels based on the different scales that the research needs to focus on: macro-scale(greater than 10^{-2}m), meso-scale(10^{-2}m～10^{-6}m),and at the micro scale(10^{-6}m ～ 10^{-10}m).In the research of solid propellant, the research on the internal grain structure of solid rocket motor and related standard test pieces belongs to the macro level, which mainly analyzes the change law of the relevant macro mechanical property parameters of solid propellant under the external load, such as modulus, Poisson's ratio, maximum tensile strength and elongation. At the micro level, the main focus is on studying the evolution law of the micro structure inside solid propellants under external loads, with a focus on revealing the mechanism of action that affects the macroscopic mechanical properties of solid propellants. At the micro scale, the changes in certain properties of solid propellants affected by them are studied at the atomic and molecular levels. This article mainly reviews the current research status of macroscopic mechanical experiments and microscopic experiments of solid propellants.

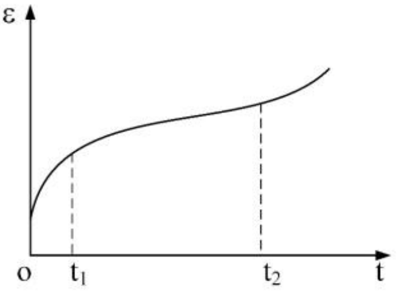
2. Current Status of Macromechanical Experimental Research on Composite Solid Propellants

At present, research on the macroscopic mechanical properties of solid propellants is mainly based on tensile and compressive tests. According to the number of loading axes, tensile and compressive tests can be divided into uniaxial tension/compression and multi axial tension/compression. According to the range of loading rates, it can be divided into creep and relaxation tests with strain rates less than 10^{-4} s^{-1}, quasi-static tests with strain rates ranging from 10^{-4} s^{-1} to 1 s^{-1}, dynamic loading tests with strain rates ranging from 1 s^{-1} to 10^4 s^{-1}, and impact tests with strain rates greater than 10^4 s^{-1}. The development of uniaxial related mechanical performance testing methods and related testing equipment for solid propellants has been relatively mature, but there is currently no standard testing method or configuration of test specimens for biaxial related mechanical tests.

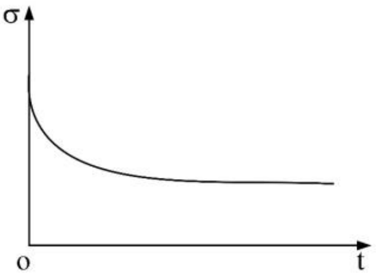
2.1. Current Status of Uniaxial Mechanical Property Testing Research

2.1.1. Current Status of Creep and Relaxation Test Research

The quasi-static mechanical performance testing of common materials under different conditions is still applicable in the field of studying solid propellants, and is currently the main means of testing the mechanical properties of solid propellants.For the uniaxial Tensile testing of solid propellants, the corresponding standards have been established at home and abroad. In China, the mechanical properties of standard dumbbell shaped test pieces made of solid propellants are mainly tested by referring to the relevant standards in GJB 770B-2005 Test Methods for Propellants and Explosives, while in foreign countries, the test methods for initiating explosive devices are tested by referring to the relevant standards in the Safety Standards for Ammunition and Explosives formulated by the U.S. Department of Defense.However, the current uniaxial testing of solid propellants cannot fully reflect the multi axial stress state and true deformation characteristics of solid rocket motor grains; During the storage period of a solid rocket motor in its full life cycle, due to the viscoelastic properties of the solid propellant itself and the effect of gravity, its internal charge will undergo creep, relaxation and other mechanical behaviors, which will lead to deformation of the internal grain of the motor, which will affect the Internal ballistics performance of the engine. This phenomenon is more serious for engines with large charges, So the creep and relaxation mechanical behavior of solid propellants are important factors affecting the structural integrity of solid rocket motors. Creep is a process in which a material undergoes slow and permanent deformation over time under constant stress conditions, and is the result of a prolonged period of action below the yield strength of the material; Relaxation is a process in which the stress level inside the specimen gradually decreases over time under constant strain conditions.



creep process



relaxation process

Figure 1. Creep and relaxation process

Li Dong et al. [1] conducted creep tests on double base propellants at different temperatures and stress levels in their study of nonlinear creep characteristics, and found that the propellants exhibited significant rate and temperature correlations. Zhang et al. [2] conducted creep tests on double base propellants under different stress levels and verified the above characteristics using several different creep models.Similarly, Shekhar et al. [3] studied the changes in mechanical properties of several solid propellants within the range of -50 to 55 ℃ and found that the elastic modulus and tensile strength of solid propellants increase with the decrease of temperature. The main difference between propellants is that their elongation trends and maximum values vary with temperature, indicating that solid propellants have a significant temperature correlation.Bihari et al. [4] used the Kelvin Voigt model to study the viscoelastic properties of HTPB propellants under different stress and temperature loads,The creep characteristic curve of HTPB propellant was obtained using Dynamic Mechanical Analyzer (DMA) during the experiment.Wang Xin et al. [5] conducted a numerical analysis of the creep process of the internal charge of a solid rocket engine under vertical long-term storage conditions by studying the creep tests and numerical simulations of the creep process of standard dumbbell shaped specimens made of solid propellant under different stress levels. After verifying the accuracy of the simulation results, they conducted a numerical analysis.

When studying the relaxation behavior of solid propellants, Prony series is usually used to fit the relaxation curve and obtain the performance characterization under the relaxation behavior. The Prony series expression is as follows:

$$E(t) = E_{\infty} + \sum_{i=1}^{n} E_i \, exp(-\frac{t}{\tau_i})$$

Where t is the time, E_{∞} is the equilibrium modulus, E_i and τ_i are the modulus and relaxation time of the i-th sticky pot in the generalized Maxwell model.

2.1.2. Research status of uniaxial tension/compression/shear/tension shear tests

At present, the research on mechanical properties of conventional materials is mainly based on uniaxial tensile/compressive/shear tests, which is also the main research method and means for studying the mechanical behavior of solid propellants. In order to obtain the mechanical property parameters related to the solid propellant, the Stress–strain curve of the solid propellant is obtained through the uniaxial tensile test of the corresponding standard specimen made of the solid propellant. Generally, the stress-strain relationship of the solid propellant is divided into three stages, namely, the linear elastic segment, the damage segment ("dehumidification" between particles and nucleation of matrix microcracks) and the damage segment, as shown in Figure 2.

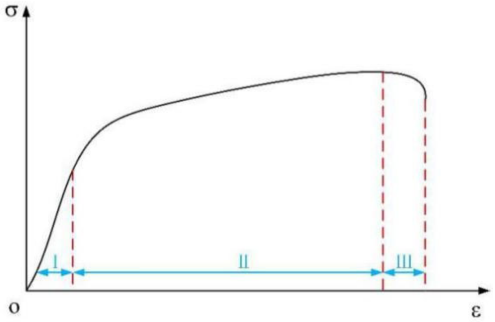


Figure 2. Typical mechanical property curve of uniaxial tension

At present, research on solid propellants generally believes that there is no damage generated inside the propellant during the online elastic stage, and there is a linear relationship between stress and strain; As the load increases, the interior of the propellant mainly begins to undergo "dehumidification" between the matrix and AP particles, leading to the formation of damage. At this point, there is a non-linear trend in the macroscopic tensile curve; As the load further increases, the "dehumidification" between the matrix and particles will converge to form microcracks, which will further converge with the increase of load, leading to macroscopic fracture failure of the propellant.

Wang Zhejun et al. [6] conducted research on HTPB propellants under low temperature and high strain rate uniaxial loading, and found that the temperature and strain rate of HTPB propellants have a significant impact on their mechanical properties. Wang Hugan et al. [7] found in their research on the low-temperature mechanical properties of HTPB propellants at different strain rates that they used the fast and slow combination tensile test device to conduct tests, as shown in Figure 3, and completed the drawing of the Stress–strain curve of HTPB propellants through the relevant data obtained from the test results. At the same time, they also found that the "double peak" phenomenon in the obtained curve would become more obvious with the increase of the tensile rate.

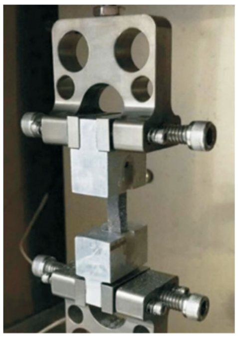


Figure 3. Fast and slow combination tensile testing fixture

Ma Hao et al. [8] studied the "dehumidification point" of HTPB propellant and the tensile strength of fast and slow groups. Through quasi-static uniaxial Tensile testing, it was found that the increase of tensile rate and the decrease of experimental temperature would significantly increase the tensile strength and other mechanical property parameters of HTPB propellant. Gong Shijun et al. [9] carried out dynamic Tensile testing of solid propellant in the range of 0.05~150s^{-1} tensile strain rate at low temperature of -50 ℃, and found that the tensile strength of solid propellant will increase with the increase of tensile rate. When the tensile rate is greater than 0.12s^{-1}, the obtained Stress–strain curve will show a "double peak phenomenon". At the same time, the phenomenon is more obvious with the increase of tensile rate. Qiang Hongfu's team [10] [11] conducted a study on the mechanical properties of solid propellants under different temperature strain rates under uniaxial tension. Yang Long et al. [12] used a universal testing machine to study the mechanical behavior of HTPB propellants under uniaxial tension, and found that the tensile strength and elongation of HTPB propellants increase with the increase of strain rate.

Blumenthal et al. [13] conducted compression tests on solid propellants under low temperature quasi-static conditions using traditional material testing machines. Ren Ping et al. [14] conducted tensile and compression tests on Rectangular cuboid solid propellant specimens of different sizes, and compared and analyzed the failure morphology of the specimen cross-section. Hu Shaoqing et al. Sun Chaoxiang et al. [16] used screw pressing technology combined with machining methods to produce cylindrical compression test pieces of solid propellants, and conducted compression characteristics tests on them at high and low strain rates. They found that their mechanical properties would change with the direction of the specimen. Therefore, samples were taken along the axial direction of the propellant column to prepare test specimens.Chang Xinlong et al. [17] used a SHPB device (split Hopkinson compression rod device) to conduct compression tests on HTPB propellants at strain rates ranging from 700 to 2050 s^{-1}.Sun et al. [18] combined a constant temperature device with SHPB to study the mechanical behavior of solid propellants under uniaxial compression at different temperatures and high strain rates. Wang Zhejun et al. [19] conducted a study on the temperature dependence of HTPB propellants under uniaxial compression and found that the compression modulus and strength limit of HTPB propellants exhibit a linear double logarithmic increasing trend with the decrease of temperature.Yang Long et al. [20] studied the uniaxial mechanical properties of carboxylic and hydroxybutyric propellants within the strain rate range of 1.7×10^{-4}～1.7×10^{-1} s^{-1}.Yang [21] and Zhang [22] used SHPB (Split Hopkinson Pressure Bar Device) to study the uniaxial compressive mechanical properties of HTPB propellants at strain rates above 100s^{-1}. Chen Xiangdong et al. [23] conducted a study on the impact performance of HTPB propellants under uniaxial compression under high strain rate loading at different temperatures.

At present, the relevant standards for conducting shear tests on solid propellants have not been established, and shear tests can more intuitively study the relevant mechanical properties of solid rocket motor grains under shear. The current research methods for conducting shear tests on materials can be roughly divided into three categories: tensile and compressive notch shear test methods, torsional cylindrical shear test methods, and bending short beam shear test methods.

The failure mechanism of solid propellants under composite loading conditions is more complex than that under single loading conditions. It is generally achieved by using a universal material testing machine in conjunction with corresponding test fixtures. When the loading direction is at a certain angle with the cross-section of the shear specimen, the load can be divided into shear load parallel to the cross-section and tensile and compressive load perpendicular to the cross-section direction. The multi ratio tension shear disc fixture designed by Arcan is widely used in tension shear experiments [24] [25] [36]. Luo et al. [27] achieved a combination of tensile and shear loading based on uniaxial loading by loading diagonally bonded specimens on a uniaxial tensile machine, thereby measuring the tensile and shear strength of the coated foil interface. Wang Jiaxiang [28] realized the low-temperature dynamic shear test research on HTPB propellant through the designed tension shear composite loading fixture combined with the uniaxial Tensile testing machine, and completed the establishment of the low-temperature Shear strength criterion for HTPB propellant.

2.2. Current Status of Research on Multiaxial Mechanical Performance Testing

Research at home and abroad shows that the inner hole surface of solid rocket motor grain is usually the part with lower Factor of safety [29] [30], and the stress state of this part is generally considered to be the state of biaxial tension [31]. Many scholars at home and abroad have made relevant experimental studies on propellant specimens under multiaxial stress state, In the 1960s, foreign researchers conducted relevant research on the mechanical properties of solid propellants under multiaxial stress states; Some domestic researchers also conducted relevant research on the mechanical properties of solid propellants under biaxial stress in the 1990s. At present, there is still relatively little research on solid propellants under multi axial stress, and there is no unified standard for the experimental methods and specimen configurations for conducting multi axial experimental research. At present, there are three main test methods for solid propellant under multiaxial stress state: quasi biaxial test based on Flat noodles specimen, biaxial Tensile testing based on cross specimen and triaxial confining pressure test based on circular specimen. The status quo of propellant multiaxial test research is summarized from these three aspects.

2.2.1. Research status of quasi biaxial mechanical performance tests

In the quasi biaxial research of solid propellant, researchers at home and abroad carry out relevant research through Flat noodles type specimens. According to the research of Bills et al. [32], processing solid propellant into Flat noodles specimens can achieve the stress state of biaxial tension in the central test area of Flat noodles type specimens, The experimental results obtained can be used to analyze the structural integrity of solid rocket motor grain under typical load conditions. The schematic diagram of Flat noodles specimen is shown in Figure 4. Wang Zhicun [33] carried out finite element analysis on solid propellant Flat noodles specimens, and the results showed that the central area of the Flat noodles specimen presented a plane strain state, which was similar to the stress state on the surface of the inner hole of the solid rocket motor grain. Renganathan et al. [34] carried out uniaxial Tensile testing on Flat noodles specimens, and concluded that the stress ratio in the central area will be 1:2 biaxial stress state. In recent years, Qiang Hongfu's team [11] [35] carried out the dynamic quasi biaxial tensile mechanical property test research on HTPB propellant Flat noodles specimens for the first time. Zhao Wencai et al. [36] carried out a study on the mechanical properties of aged HTPB propellant Flat noodles specimens under low temperature dynamic loading. Liu Chang et al. [37] [38] carried out a study on the thermal aging performance of HTPB propellant Flat noodles specimens under low temperature dynamic quasi biaxial tension. The results showed that the superposition of multiple loads would make the meso damage of propellant more serious, but the damage was weakened compared with that under uniaxial tension.

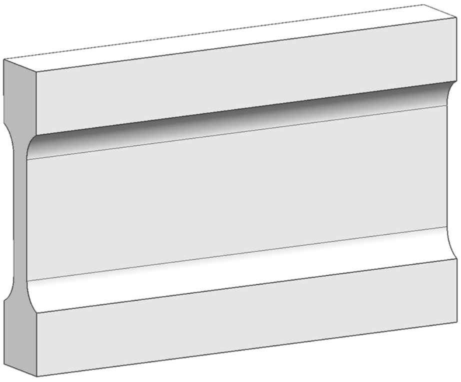


Figure 4. Schematic diagram of flat noodles test piece

The quasi biaxial test method based on Flat noodles test pieces is mainly to utilize the structural specificity of Flat noodles test pieces, but Flat noodles test pieces can only show a certain proportion of biaxial tensile stress state within a certain range of the central part, and need to be within a certain range of linear elastic deformation. At the same time, there is also the situation that different proportions of tension cannot be achieved for the same size of test pieces, It is often necessary to change some geometric dimensions of the test piece to achieve the effect of variable proportion loading, which also results in the need to redesign and determine the size and configuration of the test piece when making certain proportion of Flat noodles test pieces, which often costs a lot of time, which also brings great inconvenience to the biaxial test.

2.2.2. Current status of biaxial mechanical performance testing research

In the current research on biaxial mechanical performance testing of solid propellants, due to the unique nature of the test itself, the test specimens often adopt a cross configuration, which requires the design of corresponding supporting fixtures; In the early stages of testing the biaxial mechanical properties of solid propellants, due to the immature development of biaxial loading equipment and high testing costs, uniaxial material testing machines were mainly used in conjunction with corresponding fixtures to conduct experimental research on biaxial loading of solid propellant specimens. Zhang Lihua et al. As shown in Figure 6, Qiang Hongfu's team [40] conducted a biaxial Tensile testing on HTPB propellant through a bathtub specimen made by central thinning. Cheng Shu et al. [41] carried out a biaxial Tensile testing test study on solid propellant containing mode I cracks, and found that the driving force of crack growth must be affected by the stress parallel to the crack, as shown in Figure 7. Jia Yonggang et al. [42] optimized the design of the cruciform specimen by numerical simulation of the cruciform specimen used in the biaxial test, and obtained the biaxial tensile stress strain failure curves under different tensile rates by preparing the designed HTPB propellant specimen for biaxial Tensile testing. The results show that HTPB propellant has obvious biaxial weakening effect, as shown in Figure 8. Jalocha et al. [43], a foreign researcher, believed that the method of slotting on the arm of the cruciform specimen and thinning in the central area of the specimen could not effectively characterize the mechanical behavior of solid propellant under biaxial tension. Therefore, Jalocha et al. did not make central thinning and slotting on the arm of the cruciform specimen when conducting the biaxial Tensile testing, and finally analyzed the relationship between the viscoelastic behavior of solid propellant and strain, as shown in Figure 9. Dai Li Tu [44], in combination with the new uniaxial tensile machine and the designed biaxial Tensile testing fixture, carried out the proportional biaxial Tensile testing of HTPB propellant at low temperature and high strain rate, as shown in Figure 10. Wang Qizhou conducted a study on the mechanical properties of HTPB propellants under low temperature and wide strain rate loading on the basis of Li Tu's research.

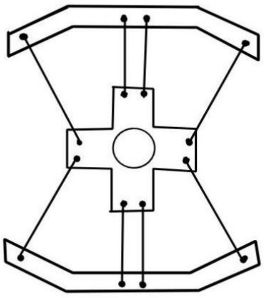


Figure 5. Right angle cross shaped specimen test

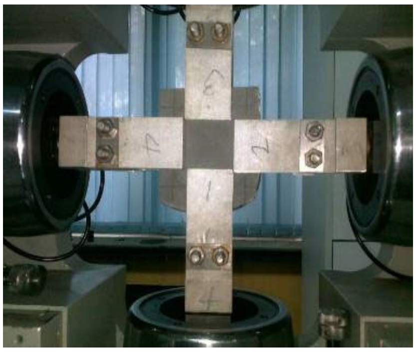


Figure 6. Bathtub type specimen test

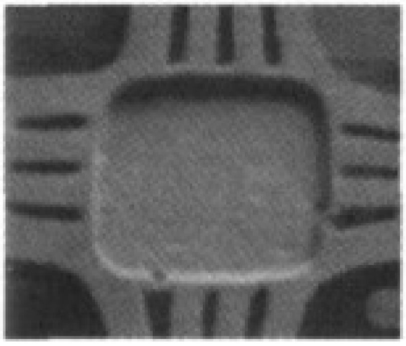


Figure 7. Cross Shaped specimen with type I crack

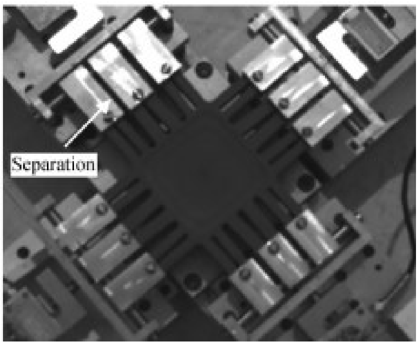


Figure 8. Cross shaped specimen with center thinning of slot on arm

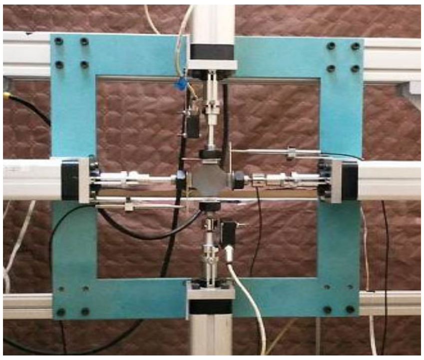


Figure 9. Unthinned fillet transition cross shaped specimen

Figure 10. Center thinning rounded transition cross shaped specimen

There is relatively little research on biaxial compression tests of solid propellants both domestically and internationally. Zhang Ya [46] carried out biaxial compression tests on the prepared Rectangular cuboid HTPB propellant specimens, and established strength criteria under biaxial compression based on the test results. Geng Tingjing [47] [48] conducted biaxial compression tests on HTPB propellants using a designed cube specimen and a biaxial compression fixture combined with a uniaxial testing machine.

2.2.3. Research status of triaxial confining pressure test

Foreign researchers Lindsey et al. [49] conducted "poker chip" experiments on circular thin plates in the 1960s to study the mechanical behavior of solid propellants under triaxial stress. Afterwards, Jones et al. [50] conducted radial compression tests on solid propellant thick disk specimens. Traissac et al. [51] conducted confining pressure tests and found that the confining pressure environment is beneficial for improving the tensile strength and elongation of propellants. Sanal et al. [52] conducted pressure tests on circular tube specimens, achieving a multi axial stress state for the HTPB propellant. The creep characteristics and relaxation modulus of the HTPB propellant were obtained through the experimental results. Liu et al. [53] conducted confining pressure tests on propellants, He Tieshan et al. [54] conducted confining pressure tests on aged NEPE, and Zhang Ya [46] conducted confining pressure tests on HTPB propellants, both of which obtained similar conclusions as Traissac et al. Zhang et al. [55] conducted a study on the influence of different confining pressures on the mechanical behavior of solid propellants under triaxial stress state based on a quasi-static testing machine combined with an adaptive confining pressure device. Zhang Jianbin [56] conducted uniaxial compression tests on double base solid propellants under different confining pressure conditions based on a confining pressure device, and obtained the variation law of mechanical behavior of double base propellants with confining pressure conditions. Shen Zhibin et al. [57] built a confining pressure test environment based on the uniaxial Tensile testing, carried out research on the change law of the mechanical behavior of HTPB propellant under the conditions of wide temperature and wide strain rate, and found that the strength of HTPB propellant will be improved under the condition of confining pressure, and the loading rate will affect the elongation of HTPB propellant. Zhang Jiye et al. [58] conducted a study on the mechanical behavior of N15 propellant under confining pressure, and found that confining pressure conditions can reduce the degree of "dehumidification" of the propellant, and the aggregation and propagation of microcracks will be inhibited by the effect of confining pressure. Li et al. [59] [60] [61] conducted confining pressure tests on NEPE propellants under different confining pressures and strain rates. The results showed that the confining pressure conditions suppressed the initiation and evolution of damage to NEPE propellants, and the confining pressure and strain rate had a significant impact on the mechanical properties of NEPE propellants. Wang et al. [62] conducted a study on the thermal aging performance of HTPB propellants under confining pressure conditions using a designed confining pressure thermal aging device. The results showed that confining pressure and thermal aging conditions significantly affect the mechanical properties of HTPB propellants. Li Chuntao et al. [63] conducted a study on the uniaxial tensile mechanical properties of four component HTPB propellants under wide temperature and strain rate confining pressure conditions. The analysis of the results suggests that confining pressure can suppress the generation of internal pores in the propellant, thereby affecting damage evolution. High confining pressure can suppress the "dehumidification" between particles and matrix, thereby weakening the influence of temperature.

3. Current Status of Microscale Experimental Research on Composite Solid Propellants

From a structural perspective, solid propellants are a multiphase structural system in which both discrete and continuous phases coexist. The AP and Al solid particles in the components are distributed as dispersed phases in the continuous phase polymer binder matrix [64]. Therefore, at the micro scale level, they are a non-uniform anisotropic material [65] [66], resulting in their macroscopic mechanical properties strongly relying on their micro structure. The correlation between the macroscopic mechanical properties and internal microstructure of solid propellants has always been an important topic that people are constantly studying [64]. Mastering the mechanism of the microscopic damage evolution of solid propellants is of great significance for predicting their macroscopic mechanical properties and guiding the improvement of the structural design of the internal charge of solid rocket motors.

3.1. Research status of microstructural observation methods

Observing the microstructure of solid propellants through experiments can help determine the qualitative and quantitative relationship between certain microstructure parameters and macroscopic mechanical performance parameters, and can also provide strong data support for the establishment of microstructure models. At present, the technical equipment used for conducting microstructural observation and research on solid propellants mainly includes optical microscopy (OM), scanning electron microscopy (SEM), ultrasonic technology (UT), computed tomography (CT) Synchrotron light source (SRS) and Nuclear Magnetic Resonance Imaging (NMRI).

Zeng Jiaya [64] observed the microstructure changes of HTPB propellant under tensile load using scanning electron microscopy, and believed that the "dehumidification" between particles and matrix was the main factor affecting the mechanical properties of the propellant. Wang Yaping et al. [67] observed and analyzed the cross section of HTPB propellant at different tensile rates based on scanning electron microscope, and found that at a lower rate, the destruction of propellant is mainly the "dehumidification" between AP particles and the matrix, and at a higher tensile rate, the destruction of propellant is mainly the matrix tearing. Ide et al. [68] conducted a comparative analysis of the cross sections of non aged and accelerated aged propellants using scanning electron microscopy, and found that AP particle fracture occurred in the cross sections of aged propellants. Chen Yu et al. [69] conducted in situ observation of the micro damage of NEPE propellant under uniaxial tension using scanning electron microscopy, and conducted quantitative analysis of the micro damage using the method of fractal dimension. They found that as the load increased, the fractal dimension increased, reflecting that the damage of the propellant was also intensifying. Liu Zhuqing et al. [70] analyzed the micro damage of propellants under different tensile strains using scanning electron microscopy and established a micro model for simulation analysis based on the observation results. Qiang Hongfu's team [38] [71] [72] [73] studied the HTPB propellant under low temperature and high strain rate loading using scanning electron microscopy, and found that under these conditions, the microstructure damage of the propellant mainly manifests as the fracture of AP particles. Yang Ming et al. [74] observed and studied the interface of HTPB propellant by uniaxial in-situ Tensile testing based on SEM. Li Gaochun et al. [75] [76] [77] [78] combined scanning electron microscopy and DIC methods to observe and analyze the adhesive interface under tensile load, and found that the strain at the adhesive interface along the tensile direction is the most significant. Liu Xinguo et al. [79] observed the cross-sectional morphology of HTPB propellant after low-temperature dynamic stretching using scanning electron microscopy, and analyzed the degree of damage using the method of fractal dimension values.

Ultrasonic testing technology utilizes sound waves with frequencies higher than 20kHz to generate different acoustic changes when propagating inside a material, thereby achieving non-destructive testing of the internal structure of the material. It has been widely used in the detection of material structural defects and can be used for the detection of metal and non-metallic materials. Knollman et al. Cobb [81] achieved the detection of particle "dehumidification", cracks, and elastic properties inside solid propellant billets based on three ultrasonic technologies. Zhao Peng et al. [82] achieved defect detection of solid rocket motor cladding and propellant based on ultrasonic water immersion detection method. Liu Jiatong [83] proposed a non-contact ultrasonic water immersion detection method based on the combination of longitudinal and transverse waves when studying the problem of interface debonding detection in solid rocket motors, and conducted interface detection on solid rocket motors.

For optical microscopes and scanning electron microscopes, the micro morphology of the surface of solid propellants can be directly observed, but it is not possible to effectively observe the micro structure inside the propellant. Composite materials such as solid propellants may experience certain surface damage under external factors other than experimental conditions, which may affect the observation results, The observed surface phenomena cannot fully reflect the accurate interaction between internal components during the experiment. Although ultrasonic technology can quantitatively describe the degree of damage in real-time, its detection accuracy may be limited when detecting solid propellants with complex microstructure. And computer tomography recognition technology (CT) can effectively compensate for the shortcomings of these observation methods.

Computer tomography (CT) technology can scan the interior of an object through the generated X-rays, and after reconstruction, obtain a true and accurate microstructure of the sample. In the study of solid propellants based on CT technology, Collins [84] [85] and Lee [86] were the first to use micro CT to obtain the three-dimensional microstructure of solid propellants, and to reconstruct and analyze the crack propagation process of propellants during uniaxial tension. Ma Changbing et al. [87] conducted scanning analysis on the microstructure of HTPB propellant based on micro CT, and obtained two-dimensional cross-sectional images of the propellant and three-dimensional spatial distribution of particles. Wang Guang et al. [88] used micro CT to obtain the micro morphology of the propellant/lining/insulation interface, and established a micro numerical model to analyze the effect of aging time on the failure mode of the bonding interface. Guo Xiang et al. [89] conducted scanning research on the interface of NEPE propellant based on micro CT, and found that scanning images can identify solid particles with different fillers on the NEPE interface. Liu Xinguo et al. [90] conducted in-situ tensile scanning analysis of HTPB propellants based on micro CT and conducted statistical analysis of the three-dimensional microstructure of the propellants. Li Shiqi et al. [91] scanned the microstructure of HTPB propellant based on micro CT and quantitatively characterized the defects in the microstructure. Li Chuntao et al. [63] conducted in situ scanning tests on four component HTPB propellants and analyzed the internal microscopic reasons for the development of macroscopic mechanical properties.

The Synchrotron light source is a Synchrotron light source, which is formed by the electromagnetic wave emitted in the speed direction when the electron moves at a high speed in the Synchrotron. It has the advantages of high brightness (high radiation intensity), wide band and low pulse. Chen Bo et al. [92] used small angle scattering technology of Synchrotron light source to test TATB insensitive explosive, and obtained its internal micro hole size and other parameters. Yeager et al. [93] observed the polymer bonded explosive with Synchrotron light source, and obtained a meso structure image with a resolution of 2 μm in 5s exposure time, which can observe the meso information such as interface, crack, pore, etc. Wang Long et al. [94] realized the three-dimensional characterization of the internal microstructure of solid propellant with the help of Shanghai Synchrotron light source (SSRF).

Nuclear magnetic resonance is mainly caused by the spin motion of atomic nuclei. Nuclear magnetic resonance imaging technology utilizes atoms to be excited by radio frequency pulses in an external strong magnetic field, resulting in nuclear magnetic resonance phenomenon. Through spatial encoding technology, nuclear magnetic signals that are detected by detectors and converted into electromagnetic signals are received, and processed by computers to form various structural images. Maas et al. [95] observed the microstructure of solid propellants based on nuclear magnetic resonance imaging. Kang Ying et al. [96] conducted experiments on the solution of HTPB propellant using nuclear magnetic resonance imaging technology and obtained the changes in the linear structure of the polymer matrix in the propellant during aging.

In summary, several methods for observing the microstructure of materials have their own advantages and disadvantages. Although optical microscopy and scanning electron microscopy can observe the intuitive and real microstructure of materials over a large range, they are limited to observing the surface of the material and cannot observe the internal situation of the material; Compared with optical microscope and scanning electron microscope, CT technology and Synchrotron light source have the advantage of higher magnification, which can observe the micro structure inside the material. The reconstructed image has a clear sense of reality and stereoscopy. Compared with CT, Synchrotron light source has faster imaging speed and stronger ability to distinguish spatial details; Although the resolution of nuclear magnetic resonance imaging technology is similar to that of CT technology, it is currently difficult to popularize nuclear magnetic resonance imaging technology in the study of the microstructure of solid propellants. In a word, the observation of propellant damage process using micro CT and Synchrotron light source can not only intuitively and accurately present the detailed characteristics inside the structure, but also quantitatively characterize the damage evolution process through relevant parameters, which has obvious advantages for the research of solid propellant microstructure.

3.2. Research status of experimental methods for microstructural observation of solid propellants under loading conditions

The research of microstructure observation test of solid propellant under loading conditions is often based on uniaxial in-situ Tensile testing combined with optical microscope, scanning electron microscope or CT and other equipment. Yang Ming et al. [74] observed and studied the interface of HTPB propellant based on uniaxial in-situ Tensile testing and scanning electron microscope; Chen Yu et al. [69] conducted in situ observation of the micro damage of NEPE propellant under uniaxial tension using scanning electron microscopy; Based on optical microscopy and scanning electron microscopy observation methods (such as fractures, sections, etc.), the spatial morphology of micropores generated by dehumidification cannot be obtained, let alone in situ damage research. With the development of observation technology, computer tomography technology can observe the interior of materials, analyze internal defects, and combine with adaptive in-situ loading devices to achieve visual in-situ observation of the damage evolution process of materials; Liu Xinguo et al. [90] conducted in-situ tensile scanning analysis of HTPB propellants based on micro CT, and conducted real-time statistical analysis of the three-dimensional microstructure evolution of the propellants under uniaxial tensile load; Li Chuntao et al. [63] conducted scanning tests on the fracture surface of four component HTPB propellants after in situ tension based on micro CT, analyzing the internal microscopic reasons for the development of macroscopic mechanical properties; Li Shiqi et al. [97] conducted a micro CT study on the micro damage of HTPB propellants under uniaxial tensile load.

Based on micro CT equipment, conducting microstructural observation experiments on solid propellants under biaxial and triaxial in-situ loading to study the microstructural damage evolution process of solid propellants under complex stress states. Currently, due to limitations in experimental equipment and technical means, research on the microstructural evolution process of solid propellants under complex stress states has not been conducted domestically and internationally, compared to uniaxial experiments, The complex stress state presented in multi axis tests is closer to the actual stress situation of solid rocket motor grain. Therefore, studying the microstructure damage evolution process of solid propellant under multi axis loading is of great significance for the structural integrity analysis of solid rocket motor grain.

4. Conclusion

(1) At present, most of the mechanical performance tests conducted on solid propellants are at the macro level. Mechanical tests based on uniaxial correlation are becoming more mature, and there is currently no unified test standard for multi-axial mechanical performance tests. Multiaxial tests are aimed at studying the mechanical behavior and damage performance of solid propellants under multi-axial stress states, and are closer to the true stress state of the grain in the engine than uniaxial tests, However, the stress under multi axial loading is more complex, and it is more difficult to

conduct multi axial tests due to equipment limitations. Compared to uniaxial tests, it is usually necessary to redesign and prepare specimens of appropriate size and configuration to facilitate multi axial tests. This has led to the current research status of difficulty and slow progress in studying the multi axial mechanical properties of solid propellants.

(2) Compared to the macro level, there are much fewer mechanical performance tests conducted on solid propellants at the micro level, which are often limited by existing equipment and testing methods; In terms of micro observation and micro mechanical property test, they are often carried out based on observation means such as optical microscope, scanning electron microscope, ultrasonic technology (UT), computer tomography identification technology, Synchrotron light source and nuclear magnetic resonance imaging. The observation range of optical microscope and scanning electron microscope observation test can only stay on the surface of the object and cannot go deep into the object for effective observation; Micro CT and Synchrotron light source equipment can use computer tomography identification technology to observe the internal microstructure of objects through scanning, which provides a powerful experimental means for studying the evolution of solid propellant in the microstructure. Therefore, it is an important direction to carry out research on solid propellant microstructure and damage evolution process based on micro CT equipment.

(3)At present, the micro mechanical performance testing methods for solid propellants are mainly based on uniaxial loading. The situation of solid propellants under uniaxial loading cannot fully reflect the response of the micro structure of solid motor grains under complex stress states. However, studying the damage evolution of the micro structure of solid propellants under multi axial stress states is basically after completing macro multi axial loading tests, Sampling is then conducted on the cross-section of the damaged specimen, and damage inversion is performed using optical microscopy, scanning electron microscopy, or CT equipment. However, this method can only qualitatively analyze the static results of the test, and cannot quantitatively characterize certain variables of the damage process; The multi axial in-situ loading tests of solid propellants at the micro level based on computer tomography recognition technology are often limited by the equipment's function and available space, which results in a research gap in the damage evolution process of the micro structure of solid propellants based on multi axial mechanical performance tests, Therefore, conducting research on the microstructure damage evolution process of solid propellants under multi-axial stress states is crucial for the structural integrity analysis of solid rocket motor grain, and is also an important direction for studying the microstructure damage evolution law of solid propellants under complex stress states.

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