INVESTIGATION INTO THE HIGH STRAIN-RATE TENSILE DEFORMATION OF ENERGETIC COMPOSITE AT LOW TEMPERATURES WITH DIC AND DMA

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ABSTRACT

Solid propellant is a typical particulate-reinforced energetic composite. In this paper, to further study the high strain-rate (0.40~42.86 s^{-1}) uniaxial tensile deformation of hydroxyl-terminated polybutadiene (HTPB) propellant at different temperatures (233~298 K), high-speed camera and digital image correlation (DIC) method were applied for obtaining the strain fields at the area of interest during the loading process. The speckles pattern was prepared on the test samples' surface by spraying white paints and drawing black dot. In addition, dynamic mechanical analysis (DMA) experiment was conducted to find out the changes in the glass transition temperature of HTPB propellant. According to the obtained strain fields at different loading time, the tensile deformation of the test sample was further discussed. Meanwhile, the reasons for the different fracture locations of the test samples at room temperature and low temperatures were analyzed. Furthermore, based on the obtained test data, the relationships between stress and time, strain rate and time during tensile deformation were discussed. Finally, the DMA test results show that the glass transition temperature of HTPB propellant rises exponentially with the increase of loading frequency, which indicates that the propellant may be in glass transition state or glass state with the coupled effects of high strain rate and low temperature.

1 INTRODUCTION

Solid propellant is a typical particulate-reinforced energetic composite, in which metal fuel particles (for example, aluminium) and oxidizer particles (for example, ammonium perchlorate, AP) are embedded in a soft polymeric matrix. Because of the compositions, solid propellant exhibits different deformation and failure mechanisms under the various strain rates and temperatures[1]. The propellant grain is the energy source and the key structural component of a solid rocket motor (SRM). Generally, the propellant grain must be designed to withstand various loads imposed by service conditions, such as thermal stress, impact, vibration and so on[2]. Therefore, it is necessary and very important to understand the deformation of solid propellant under the above loading conditions. However, up to now, to the best of our knowledge, limited information is available on the tensile deformation of solid propellants at high strain rates (1~100 s^{-1}) and low temperatures, which affects the analysis of the structural integrity of the solid propellant grain during ignition of SRM for the tactical missiles at low temperatures[3]. In addition, due to the experimental difficulties, it is generally accepted that it is very difficult to study the tensile deformation of low-strength energetic composite at strain rates ranging from 1 to 100 s^{-1}[4]. In our previous works[5-7], the uniaxial tensile properties of hydroxyl-terminated polybutadiene (HTPB) propellant, which has been widely used in present-day SRM worldwide, over the range of temperatures 233 to 298 K and strain rates 0.40 to 42.86 s^{-1} had been first analyzed successfully. The test results indicated that the mechanical properties and fracture mechanisms of the propellant under those loading conditions are significantly different from that obtained in quasi-static (<1 s^{-1}). However, the related experimental method had not been stated completely in those works[5-7]. Moreover, for completely understanding the tensile properties of

HTPB propellant at high strain rates (1~100 s^{-1}) and low temperatures, the further investigation should be made with other test methods.

In existing literatures[8-10], there are many observation techniques to monitor the deformation of various materials under stress. Recently, optical strain measurement has been increasing applied for characterizing the properties of the loaded materials[11, 12]. The digital image correlation (DIC) method is one of the optical techniques, and it is generally based on the principle of comparing speckle pattern structures on the surface of the test sample for different loading levels[13]. Due to the advantage of the whole deformation field measurement without contact, the DIC method is widely used in experimental mechanics as an effective tool, especially for soft materials[14, 15]. Knowledge of the deformation field during loading is very useful for the analysis and discussion of properties of materials. However, the published works are very little relation to the deformation field of solid propellants under various loading conditions. Therefore, further research is need.

According to the previous research[16], the deformation properties of polymeric materials are different from each other at higher and lower temperatures than the glass transition temperature. Moreover, the deformation behaviors of polymeric materials at different temperatures and strain rates can be described and predicted with the time-temperature superposition principle[17]. However, the validity of this principle is closely related to the glass transition temperature. Meanwhile, the glass transition temperature of polymeric materials is influenced by many factors. Up to now, the effect of high strain rate on the glass transition temperature of solid propellant is almost unclear.

In this paper, the uniaxial tensile strain fields at the area of interest for the test propellant sample during the loading process were obtained with the INSTRON testing machine, high-speed camera and DIC method. According to the obtained strain fields, the high strain-rate uniaxial tensile deformation of HTPB propellant at low temperatures was further discussed. Then, the different fracture locations of the test samples at room temperature and low temperatures were analyzed. Moreover, the relationships between stress and time, strain rate and time during tensile deformation were discussed based on the obtained test data. Finally, dynamic mechanical analysis (DMA) experiment was conducted to find out the changes of glass transition temperature for HTPB propellant with the effect of the strain rate.

2 MATERIALS AND EXPERIMENT

2.1 Materials and samples

The materials used here was directly taken from the composite rocket propellant and consists of 88 wt-% mixture of AP and fine aluminium particles, 7.852 wt-% of HTPB polymer binder and curing agent, and 4.148 wt-% others. The uniaxial tensile test samples were designed as a dumbbell-salt shape, which is consistent with that in our previous works[5-7]. For a complete description of the deformation fields during loading, a random speckle on the surface of the target object is necessary. In this investigation, one side of the tensile test samples' surface was painted by spraying matt white paints firstly. Then the random pattern was produced as the area of interest by drawing black dot on the white grounding, as shown in Fig. 1. To avoid the effect of humidity, the test samples with speckles pattern were stored at dry box for 24 h prior to testing.

For DMA experiment, the samples were manufactured as rectangular with a length of 70.7 mm, a width of 7.6 mm, and a thickness of 2.8 mm.

Figure 1: A random speckle pattern produced on the surface of the test sample.

2.2 Uniaxial tensile experiment with the 2D-DIC system

The high strain-rate uniaxial tensile tests conducted with a classical testing machine INSTRON VHS 160/100-20 had been stated in our previous works[5-7], and the designed aluminium gripping jaw is shown in Fig. 2. In this investigation, a 2D-DIC system is established based on this testing machine, as shown in Fig. 3. It includes a high-speed digital CCD camera (PHOTRON-FASTCAM SA.X), a light source system (DCI-1000) and a microcomputer data processing system. The incandescent lamp, with the colour temperature of 5000~5500 K, was chosen as the modeling light of the light source system in this investigation. The resolution of the camera was 1024×1024 pixels and the image acquisition rate can be up to 12500 fps (frame per second) during the test. To improve the obtained image quality, one calibration procedure was made prior to testing by repeatedly adjusting the camera position, which is in front of the window of the testing machine.

Figure 2: The designed aluminium gripping jaw.

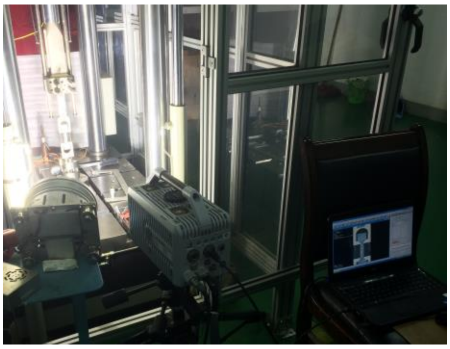


Figure 3: The 2D-DIC system based on the uniaxial tensile experiment.

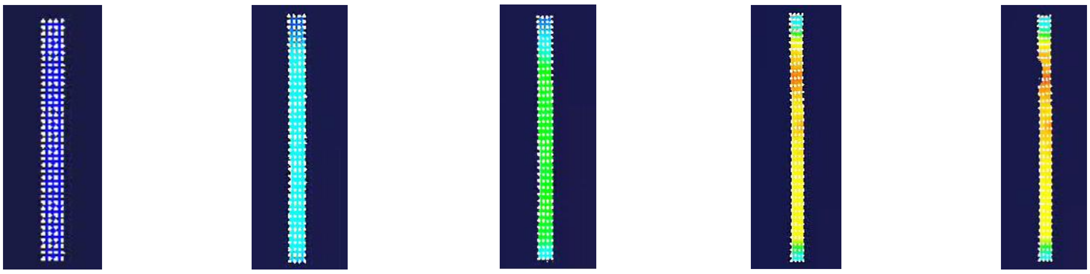
2.3 Dynamic mechanical analysis experiment

A DDV-II-EA dynamic mechanical analyzer was used to conduct the DMA experiment on the HTPB propellant. The investigated temperature range was -173 to 373 K, with heating up in steps of 1 ^{o}C/min and a soak time of 5 min. A liquid nitrogen cooling accessory was applied for the low and high temperature operations. Samples were tested at the amplitude of 5 μm and four different frequencies (3.5, 11, 35 and 110 Hz). Measurement reproducibility is very high, so only one sample was used at each test condition. During testing, the loss modulus and storage modulus were measured as a function of temperature at different frequencies, and the corresponding loss tangent was calculated.

3 RESULTS AND DISCUSSION

3.1 DIC analysis of uniaxial tensile deformation

By matching a sequence of digital speckle images captures at different loading levels in an experiment, the displacement fields of the whole loading process can be obtained. Then, the strain fields can be calculated based on these displacement fields with the microcomputer data processing system. The typical uniaxial tensile strain fields of the test propellant sample for selected steps at different loading levels are shown in Fig. 4. It can be seen that the strain fields are uniform at the beginning when testing at high strain rate and low temperature (Fig. 4(a)~(c)), however, they become heterogeneous at a later loading stage (Fig. 4(d)). Meanwhile, the stress concentration at the gripping location of the test sample is distinct, which causes the fracture of the sample at this position (Fig. 4(e)). The above phenomenon is not present when testing at room temperature. These can be explained by the following reasons. The dimensions of the aluminium gripping jaws used at room temperature were determined according to the standard dimensions of the test propellant sample. The tolerance was taken insufficient account of. The propellant becomes stiffer with decreasing temperature, thus the stress concentration at the gripping location of the test sample is more significant at low temperatures, which further induces the different fracture locations of the test samples at room temperature and low temperatures, as shown in Fig. 5. Because of these, the dimension of the gripping location for the aluminium gripping jaws was further adjusted as 11 mm instead of 10 mm at low temperatures. After improvement, the typical uniaxial tensile strain fields of the test propellant sample for selected steps at different loading levels are shown in Fig. 6. It can be seen that the stress concentration phenomenon is solved, which is helpful to get the more precise mechanical properties of HTPB propellant at high strain rates and low temperatures. In our previous works[5-7], the adjusted aluminium gripping jaws had been used.

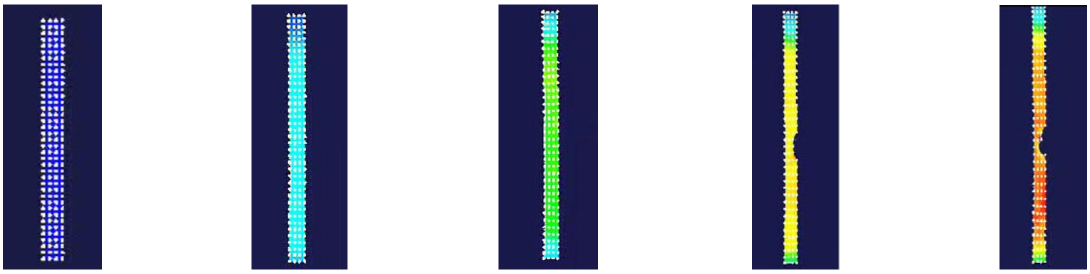


0 ms 6 ms 12 ms 24 ms 27 ms

Figure 4: Typical uniaxial tensile strain fields of the test propellant sample at 233 K and 14.29 s^{-1} with the original aluminium gripping jaws (12000 fps).

at room temperature at low temperature

Figure 5: Different fracture locations of the test propellant samples.

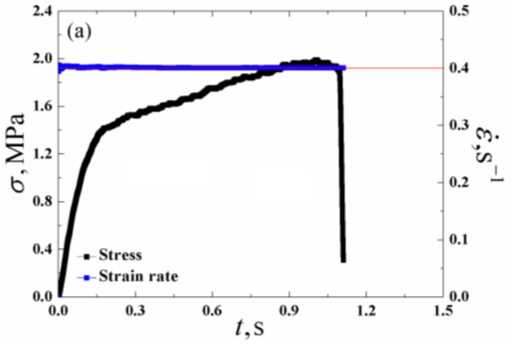


0ms 6 ms 12 ms 24 ms 27 ms

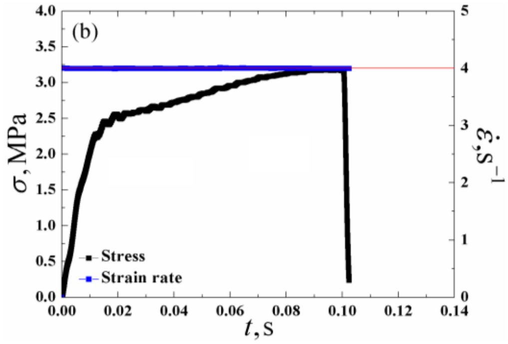
Figure 6: Typical uniaxial tensile strain fields of the test propellant sample at 233 K and 14.29 s^{-1} with the improved aluminium gripping jaws (12000 fps).

3.2 Analysis of constant strain rate

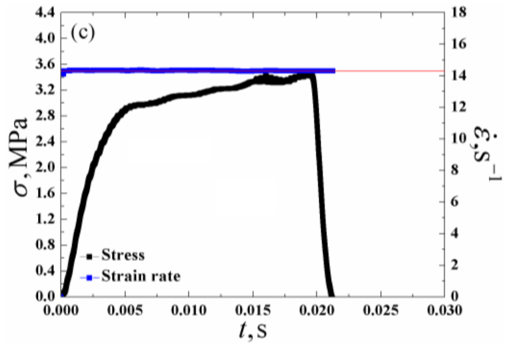
For dynamic test, the constant strain rate of the sample must be verified to ensure that the obtained characteristics of the sample were intrinsic properties of the test material. Fig. 7 presents the typical relationships between stress and time, strain rate and time during tensile deformation of the test propellant sample. From Fig. 7(a)~(c), the strain rate during tensile loading is constant at 0.40 to 14.29 s^{-1}. However, the case is more complex when testing at 42.86 s^{-1}, as shown in Fig. 7(d). Under this loading condition, strain rate is constant before the maximum tensile stress (with phase ‘AB’), then it decreases significantly when tensile stress decreasing. Because the maximum tensile stress is the initial fracture point of solid propellant, only the deformation behaviors before this point is usually considered when analyzing the tensile properties of the propellant. According to the above discussions, constant strain rate requirement can be obtained based on the test method used in our previous works[5-7].



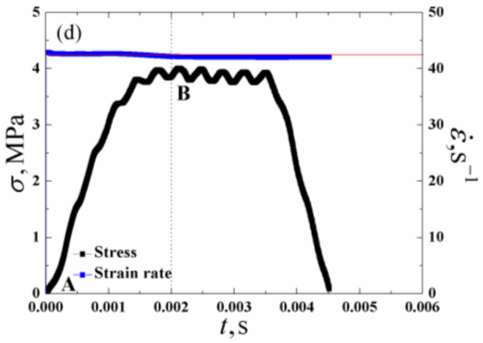
233 K、0.40 s^{-1}



233 K、4.00 s^{-1}



233 K、14.29 s^{-1}



233 K、42.86s^{-1}

Figure 7: Typical relationships between stress and time, strain rate and time during tensile loading.

3.3 Analysis of glass transition temperature

According to the test results from the DMA experiment, the variation of the glass transition temperature of HTPB propellant with the loading frequency were further obtained, as shown in Fig. 8. It can be seen that the glass transition temperature of the propellant rises exponentially with the increase of loading frequency. The higher loading frequency can cause more significant increasing of the glass transition temperature. The relationship between the glass transition temperature and the loading frequency for polymer materials can be described as follows[18]:

$$\omega = \omega_0 \exp[-\Delta E / (RT_g)] \tag{1}$$

where ω is the loading frequency, ΔE is the activation energy, R is the gas constant, T_g is the glass transition temperature, and ω_0 is the material constant.

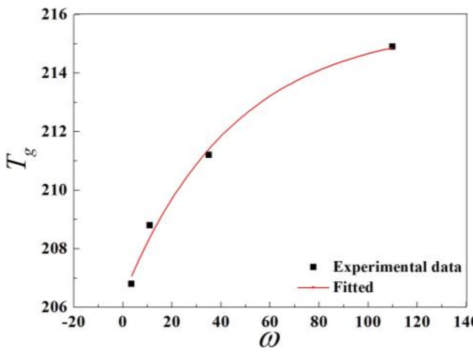


Figure 8: Variation of the glass transition temperature of HTPB propellant with the loading frequency.

Furthermore, the strain rate can be expressed with the loading frequency as[19]:

$$\dot{\varepsilon} = 2\omega(d_0 / l_0) \tag{2}$$

where ω is the loading frequency, $\dot{\varepsilon}$ is the strain rate, d_0 is the loading amplitude, and l_0 is the original thickness of rectangular sample.

From Fig. 8, Eq. (1) is suitable for the variation of the glass transition temperature of HTPB propellant with the loading frequency in this investigation. Thus, the glass transition temperature of the propellant also rises exponentially with the increase of strain rate according to Eq. (1) and (2), which indicates that HTPB propellant may be in glass transition state or glass state with the coupled effects of high strain rate and low temperature. Therefore, the mechanical properties and fracture mechanisms of HTPB propellant were influenced by the state change, which had been discussed in our previous works[5-7].

4 CONCLUSIONS

To further study the high strain-rate (0.40~42.86 s^{-1}) uniaxial tensile deformation of HTPB propellant at different temperatures (233~298 K), the DIC method and DMA experiment were successfully used in this investigation.

According to the obtained strain fields at different loading time, the tensile deformation and fracture locations of the test propellant sample at room temperature and low temperature were further analyzed. Because the stress concentration at the gripping location of the test propellant sample is more significant at low temperature, the dimension of the gripping location for the aluminium gripping jaws was further adjusted as 11 mm instead of 10 mm, which is helpful to get the more precise mechanical properties of HTPB propellant at high strain rates and low temperatures.

The analysis of strain rate during tensile loading shows that constant strain rate requirement can be obtained based on the test method used in our previous works, which further indicates that the test method proposed by us is valid for studying the tensile mechanical properties of particulate-reinforced energetic composite at high strain rates.

Based on the DMA test results, the glass transition temperature of HTPB propellant also rises exponentially with the increase of strain rate, which indicates that the propellant may be in glass transition state or glass state with the coupled effects of high strain rate and low temperature. Because of these, the structural integrity of the solid propellant grain during ignition of SRM for the tactical missiles at low temperatures may be affected.

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