Experiment research of Al particle’s ignition and combustion characteristics of NEPE propellant

Wenjie Xia\textsuperscript{1}, Yuqian Zhuang\textsuperscript{1}, Wenxiang Cai\textsuperscript{1,3}, Donglin Xue\textsuperscript{1}, Yanbang Cao\textsuperscript{1} and Xiaodong Song\textsuperscript{2}

\textsuperscript{1}Nanjing University of Science & Technology, School of Mechanical Engineering, Nanjing, 210094, P. R. China
\textsuperscript{2}Air Force Aviation University, Flight Simulation Training Center, Changchun, 130022, P. R. China
\textsuperscript{3}E-mail: caiwx_2005@njust.edu.cn

Abstract. Based on optical photography analysis method, agglomeration process of Al particles at aluminized NEPE propellant’s surface, dynamic combustion process after leaving burning surface and the collision under different pressure of N\textsubscript{2}, were photographed by high-speed camera. MATLAB was used to deal with the images to analyze the size of agglomerated particles. Experiment research results showed that higher pressure was helpful for improving ignition performance and increasing combustion efficiency of the aluminized NEPE propellant. For the agglomeration products, dimension was reduced, size distribution became more uniform, and amount of the mass increased with higher pressure environment.

1. Introduction

When the aluminum powder added to NEPE propellant increases the density of the propellant, its condensate phase products can reduce the occurrence of high-frequency unstable combustion phenomenon, but it will also reduce the combustion efficiency, erode the insulation layer and nozzle, and harm the safety of the engine [1].

Hao Hai-xia et al [2] used 50~200 nm metal powder to replace 10% normal Al powder and studied the ignition delay time of Al powder with different particle size and nanometre titanium powder by use of laser ignition. Tian Ru-yuan [3] conducted a numerical simulation of the combustion behavior of Al particles in composite solid propellants. A two-phase combustion model for the solid rocket engine was established by Liu Ping-an et al [4] and used in the flow field numerical simulation of the composited AP/HTPB/Al propellant rocket engine. Zhou Dong-hui [5] made a research about the ignition and combustion characteristics of micron/nanometre Al powder. Ao Wen et al [1] summarized the deficiencies of the existed research about Al agglomeration, and provided a valuable research direction in the next step. Liu Xin [6] studied Al particles’ agglomeration on Al propellant ‘s burning surface by use of optical shooting technology [7] and built an agglomeration model, based on the bag model [8], which can predict agglomeration particle’s size distribution. Establish a model that can predict the agglomeration granularity distribution with less computation is the trend of agglomeration model in the future [9]. Li Lian-bo et al [10] built an Al agglomeration size predicting model through fitting with the experiment data.

NEPE propellant with micron Al powder is used to research the mechanism of Al powder’s ignition and aggregation, combustion characteristics in high pressure N\textsubscript{2} environment. In order to
analyze movement state, burning process and agglomeration mechanism of Al particles on and near the surface of NEPE propellant, long focal microscope and high-speed camera are applied to observe the Al combustion process in laser ignition experiment test rig. MATLAB is used to deal with the images in order to research the influence of the pressure on the size of aggregate particles. According to the results, some methods are proposed to reduce the Al particle agglomeration and improve its combustion efficiency, which provide experiment guidance for the study of the ignition combustion characteristics and provide help for the design of the solid rocket engine and the solid rocket ramjet.

2. Experiment system and methods

2.1. Experiment system
Laser ignition experiment test rig mainly consisted of CO₂ laser generator, control system, combustion chamber and gas circuit pressure stabilization system. Horizontal laser generated by CO₂ laser generator was transferred into the combustion chamber in vertical direction by the optical path system. The pressure stabilization system connected the manual valve, solenoid gas inlet valve and solenoid exhaust valve. During the experiment, the laser power and output time were set by the computer and the pressure in the combustion chamber was adjusted by gas circuit pressure stabilization system. Figure 1 shows the experiment system used in this paper.

![Figure 1. Experiment system.](image)

2.2. Measuring devices and analytic instrument
Instruments used for measurement included high-speed camera at 1024 frames per second in 1280×1024, microlen and scanning electron microscope (SEM). Particle’s escape traces and particle diameter of the aggregate particles at different pressures were gained through dealing with certain area of the high-speed camera images by MATLAB processing code. SEM was used to scan aggregate particles combustion product.

2.3. Experiment samples
NEPE propellant is mainly composed of ammonium perchlorate (AP), nitrate adhesive, aluminum powder, et al. Solid propellant samples were processed into cuboids of 5mm×5mm×10mm as shown in Figure 2 and surfaces excluding combustion face were cladded by EPDM insulation in order to limit combustion area at a horizontal surface.

![Figure 2. Aluminized propellant.](image)

2.4. Experience methods
In this study, optical photography and high-speed camera were used to take the pictures of the Al particles agglomeration during the ignition progress at the surface of the NEPE propellant added with micron Al particles.
Before the experiment, it was necessary to calibrate the ratio of the distance to the pixel in order to calculate geometric information of the particles in the pictures captured by high-speed camera. Size calibration was applied to obtain the relationship between actual size and pixels’ number in the image. The size of the observed field could be known by reading the scaleplate. Figure 2 is used to illustrate the calibration method. There were 30 scale lines in the horizontal direction indicating that the field of view observed in the horizontal direction was 30mm, and the image resolution was 152×143, which meant the image had 152 pixels in the horizontal direction. Therefore, the actual size represented by each pixel in the image was 0.197mm/pixel. After image processing, the number of pixels representing the diameter of the agglomerate particle could be multiplied by 0.197mm/pixel to obtain the true diameter of the particle. Each pixel in the vertical and horizontal directions represents the same true size, and the image captured by the high-speed camera through the telephoto microscope reflects the shape of the object being photographed without distortion. If not, then the shape of the object on the image is not the real shape.

In particular, MATLAB processing code were used to deal with experimental image with aggregate particles, including grayscale graph conversion, valve segmentation (binary), image morphology processing, aggregate particles contour outline, center position calibration, and calculation of the maximum inner cut circle diameter and other parameters. Finally, with the centralized control, operation of the particle size and motion parameters in pixels could be converted into the desired actual characteristic in geometric dimension. This process showed that the calculation error would decrease with the increasing of high-speed camera resolution.

2.5. Experiment conditions
Pressure played an important role in Al particles ignition and combustion, especially for practical solid rocket’s combustor with the high pressure. Experiment propellant was ignited by 200W laser at 0.1MPa, 1MPa, 2MPa, 3MPa, 5MPa and 7MPa under 100% nitrogen environment.

3. Results and discussion
3.1. Combustion of Al particles
Ignition and combustion process of the propellant at different pressures nitrogen environment were photographed by the high-speed camera, and some important instants are analyzed, as shown in Figure 3.
As to the results of 0.1MPa (Figure 4), a weak reflected laser light appeared after the laser started \((t = 0\text{ms})\). After 20ms, an obvious flame could be observed. Most of the Al particles began to burn and the flame was dark yellow on the propellant surface. Part of flame near the propellant surface was brighter. As the Al particles burnt and released heat, the flame diameter and height gradually increased at 2272ms. The flame was biggest and reached the highest brightness when the combustion was the most intense. After a period of time, the energy emitted by the burning Al particles gradually decreased and the flame weakened and finally extinguished \((t = 7414\text{ms})\).

![Figure 4. Flame morphology under 0.1MPa.](image)

The time of flame spreading over propellant’s and the time of flame being the biggest decreased as the pressure increased (Figure 4 ~ Figure 7). And flame brightness and flame area increased significantly, correspondingly. When the diameter of injected Al particles became larger, combustion became fierce and combustion duration increased. It can be seen that increasing the pressure can promote the combustion of Al particles with higher intensity, efficiency and ignition combustion performance.

![Figure 5. Flame morphology under 1MPa.](image)

![Figure 6. Flame morphology under 2MPa.](image)

![Figure 7. Flame morphology under 3MPa.](image)

At different pressures, the ignition and combustion of aluminized propellant specimens are recorded with high speed cameras, as shown in Figure 8. During combustion process of the aluminized propellant, Al particles were heated and melted on the propellant surface, forming small spherical droplets. Al droplets constantly accumulated and gathered. Finally, small droplets agglomerated into larger droplets and then separated from the combustion surface. Then, the liquid aggregate particles reacted with the surrounding gas at high temperature and high pressure to form an alumina shell. Because it was continuously heated by flame and reaction heat feedback, surface temperature of Al particles exceeded the melting point, and Al droplets were wrapped in a layer of liquid oxide film and constantly expanded. Under the liquid vapor pressure, this liquid oxide film was blown up. Al vapor spread around through the liquid oxide film and reacted with the oxidant in the surrounding \([11]\).

During the combustion of Al, the Al vapor reacted with the oxidizer to form small alumina particles, and alumina smoke might collide, coalesce, or crush when moving in the air current.

Observing continuous pictures of the combustion process, collision was observed after agglomerated Al particles left the burning surface, as shown in Figure 8. Two smaller Al particles separated from the combustion surface, collided and merged to form a larger new agglomerate \([12]\).
3.2. Microstructure analysis of combustion products

Micro-structural analysis of the residual combustion products was applied to better understand Al particle’s combustion process. There is a small plate placed at the bottom of the combustion chamber is used to collect combustion products. Figure 9 and Figure 10 are SEM images of NEPE propellant combustion products under different pressures. Table 1 and Table 2 are corresponding EDS element analysis.

![Figure 8. Al particle collision.](image)

**Figure 8.** Al particle collision.

| element | wt% | Atomic percentage |
|---------|-----|-------------------|
| C       | 14.42 | 22.73             |
| N       | 5.87  | 7.93              |
| O       | 28.00 | 33.14             |
| Al      | 47.89 | 33.61             |
| Si      | 3.81  | 2.57              |
| Mg      | 0.02  | 0.02              |
| Total:  | 100.00 | 100.00            |

**Table 1.** EDS of spherical substances at 0.1MPa.

It can be observed in Figure 9 that combustion production is composed of irregular clusters of different sizes, which are substantially less than 10 μm. And the surface is relatively smooth.

The EDS analysis of the spherical material under different pressure, as shown in Table 1 and Table 2, show that it is mainly composed of Al, O and other elements, with a mass percentage of 47.89% and 28.00% respectively. According to the mass ratio of Al and O elements in Al$_2$O$_3$ is 9:8, if all the Al elements generating the corresponding oxide, 42.57% is required, thus the Al propellant combustion product contained some Al particles without combustion in addition to the alumina Al$_2$O$_3$, which indicates that the propellant combustion is insufficient. At the same time, it is found that some smaller oxide spheres are covered over some larger oxide sphere surfaces, suggesting that Al droplets aggregate particles detached from the combustion surface may expand, spray, merge, break and collide with other aggregate particles during the combustion process.
Table 2. Spectrum of spherical substances at 3MPa.

|       | C   | O   | Al  | Ca  |
|-------|-----|-----|-----|-----|
| 48-2 1| 82.04 | 13.63 | 2.23 | 2.11 |
| 48-2 2| 85.08 | 11.87 | 1.71 | 1.35 |
| Mean  | 83.56 | 12.75 | 1.97 | 1.73 |
| Sigma | 2.15 | 1.25 | 0.37 | 0.54 |
| Sigma Mean | 1.52 | 0.88 | 0.26 | 0.38 |

It is seen from Figure 10 that when the combustion chamber pressure is 3MPa, the combustion product is also composed of many spherical substances, but its surface is smoother compared to Figure 9. According to EDS analysis, it is seen from Table 2 that the mass percentage of the main element Al, O is 1.965% and 12.748% respectively, and the mass ratio of Al and oxygen is less than the Al₂O₃ which is 9:8. So the spherical material was alumina particles without excess Al particles, and the globular integrity was better, which also showed that when the combustion chamber pressure was bigger, Al aggregated particles could burn better when leaving the combustion surface.

3.3. Agglomeration influenced by the pressure

Transient pictures of agglomeration phenomenon under different pressures were processed by MATLAB code to obtain particles’ size parameters of the combustion product. Figure 11 shows the agglomeration and combustion before processing and after processing under 0.1MPa. And Figure 12 shows that under 1 MPa. It can be observed that the location information and granularity information of the burning aggregate particles can be better obtained after the combustion image processing by MATLAB. The white connected area in Figure 11 and Figure 12 is the aggregate particles, and the marked serial number of the aggregate particles is given through processing to facilitate statistical analysis. And for the too large white connected area, whether or not the aggregate particles can be identified from the real photos determines the final calculated aggregate particles.

Figure 11. Pictures processed by MATLAB under 0.1MPa.

Figure 12. Pictures processed by MATLAB under 1MPa.

4. Conclusions

Through the research of dynamic process of Al particles near the propellant surface under different pressures, following conclusions can be summarized:
Optical photography of Al particles combustion process under different pressures showed that increasing the pressure helped to improve the efficiency, the intensity of combustion.

2. Al agglomerate departing from propellant surface would collide, coalesce or break again while moving in combustion chamber.

3. With the increase of the pressure, the number of large aggregate particles decreased and the more uniform the granularity value was. But after the pressure reached a certain value, the effect of the pressure on the aggregate particles was basically saturated.

4. The microtissue structure of the combustion product of aluminized propellant was a series of irregular clusters of globular material of different sizes. With smoother microtissue structure under higher pressure, the agglomerate burnt more fully.

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