Study of aluminium nanopowder combustion by method of laser-speckle correlation

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Abstract. In this paper, we discuss an application of the method of laser-speckle correlation for studying the combustion process of aluminium nanopowder in air and its mixture of iron nanopowder. It is suggested to characterize the combustion process using the statistic parameter – correlation coefficient. The principle of observation is explained and the experimental schema for investigating is provided. The differences between aluminium nanopowder and its mixture with iron nanopowder are also considered.

1. Introduction
The innovation of modern advanced equipment and instruments is inseparable from the advancement of materials science. Because of the unique properties of new materials, their discovery can provide new development directions and performance enhancements for traditional technologies. Some of these materials are manufactured by one or several high-temperature processing procedures. Therefore, the effective monitoring and observing of these processing procedures is a key technique to guarantee the quality of materials.

The manufacture of aluminium nitride by combustion of aluminium nanopowder in air is classified as a high-temperature process [1–3]. The temperature of the combustion reaches 2500 °C. Moreover, the aluminium nanopowder is wildly used as an additive in solid propellants, thermites, and explosives to improve the energy effect of combustion. Besides, aluminium-based materials provide some potentially high impact commercial opportunities in the automotive industry and microelectronics [4, 5].

Laser speckle is a special coherence phenomenon, which is produced by coherent source of light, when it illuminates rough objects. Analysing the laser speckles is possible to observe the changing of objects and characterize these changes by some statistical parameters. In practice, many sorts of changes is possible to investigate by the laser speckle techniques. Therefore, the laser speckle technique as a new optical detection method in decades, which is currently under investigation and widely used in industry, biomedical and other fields [6–8].
2. Problem statement
The mechanisms of AlN formation by combustion of aluminum nanopowder and its mixtures in air is currently under investigation to understand the nature of the synthesis process and to control the production of ceramic powders with a specified phase composition. High temperatures, intensive background lighting and high reaction rates during aluminum nanopowder combustion make the observation of the combustion process more complicated.

Because of the superior properties of aluminium nanopowder, researchers have developed a number of methods to investigate them [9]. Using the shock tube to study of aluminium combustion is a general method [10, 11]. The shock tube can generate highly controlled elevated temperature and pressure conditions with various oxidizers that provide more possibility to understand the properties of aluminium combustion. In the work [12], the authors added several optical fibers on a shock tube to register the gas phase emission during aluminium nanopowder combustion using photodiodes connected to optical fibers. In the work [13], authors suggested to use an optical system with brightness amplification for visualization of aluminum nanopowder combustion. Ther paragraphs are indented (BodytextIndented style).

However, most of these studies depend on the particular reaction properties of aluminium nanopowders. Therefore, finding a simple way to study of nanopowders combustion is a perspective task. All nanosized metals are of dark grey or black color [1–3] and they are interesting objects for optical properties research. Therefore, using an optical method to observe the changes of aluminium nanopowder during the combustion process in air is a relatively easy and effective solution. Based on this, the method of laser speckle correlation seems to be a suitable one for aluminium nanopowder studies.

In this work, the method of laser speckle correlation is suggested to study the temporal characteristics of combustion of aluminium nanopowder and its mixture with iron nanopowder.

3. Experimental technique

3.1. Method of Laser Speckle Correlation
A laser-speckle pattern is a special combination of spots with varying brightness and irregular shape. These spots are produced by artificial spray or interference phenomenon, which is caused by coherent light. The coherent light does not require high brightness and does not damage the detected object. Therefore, the speckle pattern produced by the interference phenomenon has many application fields.

The speckle technique for observing the combustion process in this work based on the method of laser speckle correlation. The essence of this method is characterizing the changing parameter by the correlation coefficient. The correlation coefficient is calculated by the correlation formula, which is usually used for determining differences between two one or two-dimensional arrays. In this case, we used this formula to determine the difference between two adjacent speckle images, which captured using a high-speed camera.

The correlation formulas used in laser-speckle analyses were previously discussed in [14]. In this paper, we used the appropriate correlation formula (1) to calculate the correlation coefficient:

\[ C = \frac{\sum \sum [f - \bar{f}][g - \bar{g}]}{\sqrt{\sum \sum (f - \bar{f})^2} \cdot \sqrt{\sum \sum (g - \bar{g})^2}} \]  

(1)

where \( \bar{f} \) is the average intensity of the calculating area in the current speckle image; \( \bar{g} \) is the average intensity of the previous speckle image; \( f \) is the intensity of every pixel in the calculating area; \( g \) is the intensity of every pixel in previous speckle image; \( C \) is the correlation coefficient of the current and previous speckle images. This formula was used in our previous work [14].

3.2. Experimental Setup
The scheme of the experimental setup is presented in Figure 1. The light source in our experiments was an Nd:YAG laser with the variable power from 5 mW to 200 mW. It generated the coherent light
of 532 nm wavelength for achieving a prerequisite of producing the laser-speckle patterns. The diameter of the laser beam was 0.9 mm. The objects under investigation were the aluminium nanopowder sample and the mixture of aluminium and iron nanopowders. In this work, these samples were shaped into a 2 cm long aluminum nanopowder cuboid (Figure 2), and were ignited by a gas burner. The rough surface can extremely scatter the illuminating laser beam. The filter with a narrow bandpass of 532 nm wavelength was installed to pass the reflected laser speckles and isolate the background radiation light emitted by the burning sample. The high-speed monochrome camera Phantom Fastcam SA1.1 with Canon Macro Lens EF 100 mm objective lens set on infinity focus was used for capturing the speckle images.

The light path in this experimental setup is quite simple. The laser irradiated the center of the nanopowder sample. The reflected scattered light produced the speckle patterns, captured by the camera through the bandpass filter.

![Figure 1. Experimental scheme: 1 – high-speed monochrome camera with objective, 2 – 532 nm narrow-band filter, 3 – cuboid nanopowder, 4 – Nd:YAG laser.](image1)

![Figure 2. The typical views of aluminum nanopowder cuboid samples before combustion and after.](image2)

3.3. Principle of Imaging
Figure 3 provides two different laser-speckle images captured during combustion. These two speckle images show a total different speckle patterns. The difference indicates that the surface shape of the sample is changing during combustion. The velocity of the combustion process is inversely related to the similarity of adjacent laser-speckle images.

![Figure 3. Laser speckle images during the aluminium nanopowder combustion.](image3)
The aluminium nanopowder combustion in air propagates via two heat waves [13, 15]. After ignition, the first heat wave propagates through the sample with low temperature 600–800°C. During the second heat wave, the combustion process turns into a thermal explosion mode with a high combustion temperature 2000–2400°C [15]. Figure 4 shows the time dependence of the correlation coefficient for the combustion. The curve can be divided into three parts corresponding to the heat waves propagation. The first part corresponds to the time interval from ignition to the start of the first heat wave. In this part, the first wave propagates from the ignited area to the observing area. The second part record is the duration of the first heat wave, and the third part shows the duration of the second heat wave.

**Figure 4.** The schematic time dependence of the correlation coefficient during the aluminium nanopowder combustion.

4. Results and discussion
The combustion of four aluminium nanopowder samples are characterized by the correlation coefficients shown in Figure 5. All these curves, following the principle of observation, demonstrate two characteristic parts corresponding to two heat waves. The propagation time of both heat waves at the observing area can be measured using these curves. The time parameters of combustion measured using the data in Figure 5 are summarized in table 1.

**Figure 5.** Time dependences of the correlation coefficient during the combustion of four aluminium nanopowder samples.
The combustion process of aluminium and iron nanopowder mixture is characterized by the correlation coefficient shown in Figure 6. These four curves demonstrate three parts of combustion, which correspond to two heat waves. Compared with the curves in Figure 5, the second heat wave in Figure 6 starts earlier, which means that the time difference is smaller. In addition, the duration of both two heat waves in Figure 6 is smaller than for the aluminium nanopowder curves in Figure 5. These time parameters are summarized also in Table 1.

![Time dependences of the correlation coefficient during the combustion of the mixture of aluminium and iron nanopowders.](image)

**Table 1.** The time parameters of combusions.

| No. | Aluminium nanopowder | Mixture of aluminium and iron nanopowders |
|-----|----------------------|------------------------------------------|
| 1   | 1.19                 | 1.08                                     |
| 2   | 1.31                 | 1.08                                     |
| 3   | 1.32                 | 0.88                                     |
| 4   | 1.05                 | 0.88                                     |

5. Conclusions

In this work, the combustion processes of aluminium nanopowder and its mixture with iron nanopowder are studied by the method of laser speckle correlation. We obtained the time dependences of the correlation coefficient during the combustion and explained the observation principle using the correlation coefficient for characterizing propagation of the front and heat waves of combustion.

The curves of the time dependences of correlation coefficient and the time parameters, which were used for characterizing the properties of combustion, provided the capability of the method application for determination of nanopowder type. Therefore, the method of laser speckle correlation needs more in-depth research in further to define the time parameters for other kinds of mixtures of aluminium nanopowders.
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