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Study of self-propagating high-temperature synthesis of aluminium nitride using a laser monitor

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ABSTRACT

This study focused on the synthesis of aluminium nitride (AlN) by combusting aluminium nanopowder in air. To investigate the combustion of aluminium nanopowder, a copper bromide laser monitor was used. The optical system equipped with brightness amplification allowed the elimination of the background lighting effect and enabled the high time-resolved recording of the process. In particular, the laser monitor enabled us to detect changes in the morphology and optical properties of the surface of the aluminium nanopowder sample as well as to observe the propagation of the combustion waves in spite of the intense background lighting during combustion. The main time parameters of the combustion of aluminium nanopowder in air were determined. To improve and facilitate the processing of laser monitored high-speed video recordings, we proposed to analyse the time dependence of the intensity of the output signal of the laser monitor. The dependence was used to successfully detect the occurrences of all combustion waves and describe their dynamics. The time dependence also favourably represented the evolution of the reflection coefficient of the combustion products of aluminium nanopowder. This is the first time that this property of aluminium nanopowder has been investigated. The reflection coefficient evolution coupled with video recordings of the sample surface development during the combustion of nanopowder could be used to control the combustion process.

1. Introduction

Presently, a number of methods used for producing new materials are associated with the interaction between matter and powerful energy fluxes [1–14] or high-temperature combustion processes [15–19]. One of these processes is the manufacturing of aluminium nitride (AlN) by combusting aluminium nanopowder in air [20–26]. Both AlN and ceramic materials based on it are among the most promising materials used as heat-removing dielectric substrates in microelectronics [27–31] and optoelectronics [32–34].

Manufacturing AlN-based ceramic materials with required properties has been attempted by various research groups [35–37], but the mechanism of AlN formation during the combustion of aluminium nanopowder in air and the changes in the morphology of the synthesised products have not been studied enough to understand the nature of the processes and to control them. The high temperatures (~2500 °C) reached during the combustion of aluminium nanopowder and the high reaction rates complicated the studies. Therefore, there is an increasing need to develop specific research methods and techniques, especially visual control methods.

Nowadays gas lasers, in particular, metal vapour lasers are less popular than solid-state lasers. However, due to certain distinctions, some gas lasers and their active media can be expedient and can exhibit superior performance for a number of specific tasks. For instance, copper (copper bromide) vapour lasers exhibit unique characteristics such as natural narrow amplification bandwidth [38–42], high single-pass gain [42–47], high pulse repetition frequency [48–51], operation in the visible spectral region [52–55], high average power [56–61], large cross-section active media [61–67], and the ability to control laser radiation parameters [68–72]. The combination of these features in one laser renders their active media appropriate for use as brightness amplifiers in optical devices such as laser projection microscopes [73–77] and laser monitors [78–82]. The main advantage of these devices is their ability to visualise objects shielded by intense background lighting, in particular, allowing the observation of objects through flames or plasma. Laser projection microscopes and laser monitors allow the observation of objects shielded by background lighting with an intensity corresponding to the blackbody radiation intensity at temperatures that are estimated to exceed 10,000 °C [82].

To the best of our knowledge, active investigations on developing a

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laser monitor [82,83] and improving its characteristics [84–86] are currently carried out mostly in Tomsk (Russia). Unfortunately, investigations directed toward the exploration of new laser monitoring application fields are not receiving sufficient attention, and that is considerably hindering the evolution of the technique and its increase in popularity. However, achievements in developing a laser monitor as well as the availability of high-speed digital video cameras indicate the great potentialities of laser monitor applications. One such potentiality is investigated in the current work.

2. Experimental

The aluminium nanopowder used in this work was produced by electrical explosion of aluminium wire in argon using the UDP-4G setup developed at Tomsk Polytechnic University [87]. The same nanopowder is commercially available from Advanced Powder Technologies LLC [88]. The activity parameters of the aluminium nanopowder were as follows: initial oxidation temperature of 450 °C, oxidation level of 63.8%, maximum oxidation rate of 0.13 wt%/°C, and specific heat effect of 4995 J/g. The particle size distribution of the nanopowder was close to the logarithmic normal with a maximum of 120 nm. The amount of metallic impurities in the nanopowder did not exceed 1.0 wt %.

The powerful thermal radiation that was generated during the combustion of aluminium nanopowder complicated its study using conventional methods of visual control. Therefore, we suggested the use of a laser monitor to observe the combustion of aluminium nanopowder in air. The laser monitor was designed based on a compact copper bromide brightness amplifier using a semiconductor pumping source. The aperture of the gas-discharge tube was 1.5 cm and the length of the active area was 40 cm. The pulse repetition frequency of the brightness amplifier was 24 kHz, the superradiance pulse duration was approximately 40 ns, and the superradiance pulse energy was 2.7 µJ.

The scheme of the laser monitor used in this work to observe the combustion of aluminium nanopowder in air is shown in Fig. 1, and its principle of operation is as follows. The superradiance of the brightness amplifier illuminates an area of the object under observation through the lens (the size of the area depends on the lens used). The reflected light carries information about the surface of the photographed object, which is amplified while the light travels through the brightness amplifier, and it is then projected onto the camera matrix. The grey filters installed at the amplifier output interrupt the broadband thermal lighting due to the combustion of the aluminium nanopowder which is significantly weaker than the desired signal.

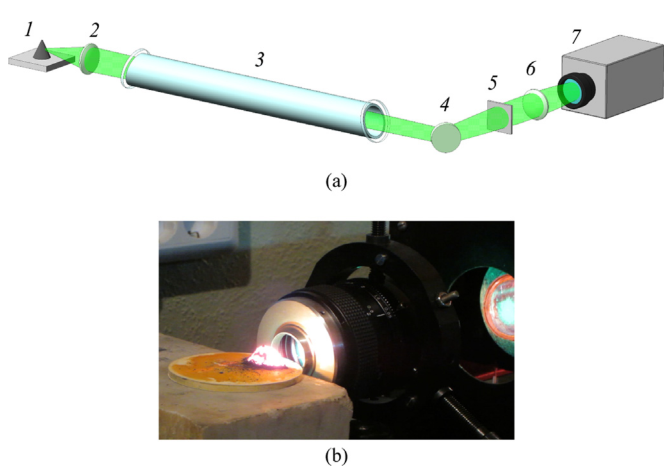

Fig. 1. (a) Scheme of the experiment and (b) photograph of the combustion process: 1 – monitored sample, 2 – image-forming lens, 3 – copper bromide brightness amplifier, 4 – turning mirror, 5 – grey filter, 6 – image-scaling lens, and 7 – high-speed camera.

The capability to visualise processes shielded by intense background lighting and to observe objects through flames or plasma using laser monitors is available due to the natural high monochromaticity of the radiation generated by the active medium and correspondingly the high degree of spectral selectivity of the brightness amplifier based on the same active medium. Thus, the total signal energy is concentrated in a narrow spectral line and a narrow time interval that provides a significantly higher signal intensity than the radiation intensity of most thermal sources at the same wavelength and for the same time interval [82].

The working wavelengths of copper vapour and copper bromide vapour active media are 510.6 and 578.2 nm. The contribution of each of the laser transitions to the total radiation intensity depends on the operating conditions of the active element of the laser [89,90]. For the copper bromide active medium used in this work, the contribution of the $4p^2P^0_{3/2} \rightarrow 4s^{2\,2}D_{5/2}$ laser transition of atomic copper significantly exceeded the contribution of the $4p^2P^0_{1/2} \rightarrow 4s^{2\,2}D_{3/2}$ laser transition, i.e. the resulting radiation of the brightness amplifier as well as the images obtained using the laser monitor were monochromatic. Practice showed that working on one of the laser transitions ensured better image quality using a copper vapour laser monitor. Thanks to the aforementioned brightness amplifier feature, no additional selective filter were required in our study.

To obtain high quality recordings for fast processes, it is highly desirable that every image should be formed by a single radiation pulse of the brightness amplifier. Thus, the brightness amplifier and the recording system should operate synchronously, i.e. there should be a strict temporal relationship between the camera exposure and the arrival of the signal amplified by the brightness amplifier. This ensures minimal distortion and maximum temporal resolution (if required), which is limited by the pulse repetition frequency of the brightness amplifier. To guarantee these requirements were met, we used a so-called laser monitor with single-pulse image recording [82,83].

Images were recorded using a high-speed HiSpec FastCam 1 video camera with available external synchronisation. The used camera had 2 GB of internal frame memory and an available minimum exposure time of 2 µs. However, to ensure the best contrast for the images obtaining using such an optical system, it would be advisable to use a high-speed video camera allowing to vary the exposure time to nanoseconds to separate the desired signal from background light more accurately.

To avoid omitting important information during the combustion of aluminium nanopowder, the video recordings in the current work were performed using different frame rates. The maximum recording speed used in the research was 1200 fps. Since the pulse repetition frequency of the used brightness amplifier was constant (24 kHz), the synchronisation of the brightness amplifier with the video camera was achieved by dividing the superradiance repetition rate of the brightness amplifier by a specified factor. The synchronisation principle is described in detail in the literature [82].

When recording a video using a higher frame rate, the duration of the video recording is restricted by the limited internal frame memory capacity of the video camera. For instance, the maximum recording time for a frame rate of 1000 fps and reduced resolution of 500 × 500 pixels amounted to 8.5 s for the high-speed digital video camera used in our study. Therefore, the available video camera restricted the performance capabilities of the laser monitor.

In this work, the video recording was manually started by the operator when the combustion front approached the area of radiation we targeted on the surface of the sample. To determine the scales of the recorded images, the optical system was previously calibrated using a microscope calibration ruler.

3. Results and discussion

In this work we investigated the combustion of aluminium