

Spectral effects in laser induced incandescence application to flame-made titania nanoparticles[☆]

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Received 28 September 2007; accepted 18 November 2007

Available online 23 November 2007

Abstract

Particle size is a crucial parameter in nanopowder use and production. A method to obtain information about this during the particle synthesis would be very valuable in order to optimize the process. In the field of nanoparticles flame synthesis, an extension of the techniques used in soot diagnostics sounds rather obvious, but it is far from being easily accomplished. In this paper investigations on the application of the laser induced incandescence to TiO₂ nanoparticles in a flame reactor are reported. The work basically concerns the effects of laser fluence on the spectral structure of the laser-excited radiation emitted from the particles. It will be shown that in many cases such a radiation cannot be plainly assumed as an incandescence signal. Measurements of the signal time decay are reported and interpreted in the light of the spectral indications. Finally, the dependence of such a time decay on the particle size is experimentally demonstrated.

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Keywords: Laser induced incandescence; Combustion diagnostics; Flame synthesis; Titania nanoparticles

1. Introduction

Nanoparticles are produced in a number of ways (e.g. milling, sol–gel, ablation–coagulation) among which flame synthesis plays a particularly important role. The possibility of producing a rich variety of tightly dispersed, highly functionalized, high purity particles is actually outstanding with this technique [1–4]. Moreover, the features of continuous process and very low post-treatment make flame synthesis attractive for mass production, for which it is largely employed indeed. For instance, titania (TiO₂) is produced in tons in this way [5].

Particle size is a major issue in nanopowder characterization, use and, obviously, production. In fact, both primary particle size and degree of aggregation between primary particles are

important to determine the features of the final product. Here, the widely shared terminology aggregates=strong necks, agglomerates=weak necks between particles will be used. In particular, one of the more important parameters of nanopowders, the surface/volume ratio, basically depends on the primary particle diameter. Small particles, even though significantly aggregated or agglomerated, allow to keep high such ratio. In any case, even after their production nanoparticles tend to agglomerate to some extent beyond their original agglomeration degree. In other words, when a nanopowder is used, the agglomeration degree also depends on the material recent history, while the primary particle size does not. In addition, many powder characterization systems, such as Photon Correlation Spectroscopy (PCS) and the different types of centrifuges, are sensitive to agglomerates rather than to primary particle size. This leads to the conclusion that the availability of diagnostics enabling the access to the primary particle size monitoring and control definitely would allow to establish an important parameter.

Laser Induced Incandescence (LII), a well established method in soot diagnostics [6–8], appears as an attractive way to the goal. In such a technique, a short (typically 20 ns), intense

[☆] This article is published in a special honor issue dedicated to Jim Winefordner on the occasion of his retirement, in recognition of his outstanding accomplishments in analytical atomic and molecular spectroscopy.

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enough laser pulse heats up the particles well above the environment temperature (e.g. 4000 K for soot in a 2000 K flame) [9,10]. Therefore, particles emit a spatially, temporally and spectrally recognizable radiation that, under proper experimental conditions, follows the grey-body emission law [11]. The intensity of such a radiation lasts much longer (hundreds of nanoseconds) than the laser pulse and decreases exponentially. According to many researchers in the field, see e.g. Refs. [12,13], for loose agglomerates with point contact, the decay time basically depends on the primary particle size, while a shielding effect could arise for higher agglomeration/aggregation degrees. Although this could be an open question, the detection of the incandescence decay time (Time Resolved Laser Induced Incandescence, TIRE LII) appears to be perhaps the only way to obtain useful, almost real-time indications about the particle size during synthesis.

Applications of this kind have been already suggested or considered in perspective [14–16], have been approached both theoretically and experimentally [13,17] and some applications have been attempted [18,19]. A study on the cooling of metal particles has also been recently published [20]. Nevertheless, the literature on LII from non-soot particles, especially if compared with the one on carbonaceous particles, remains extremely poor, being largely included in what was summarized above. This situation seems to persist in spite of the fact that the publication of the cited work in Ref. [17], rather wide and promising, dates eight years back. Moreover, to our knowledge, no LII applications in flame synthesis have been described so far, although this seems to be the most straightforward extension of what is done in soot diagnostics. In a sense, this situation suggests that such an extension is not actually straightforward. Deep differences exist in fact between soot and synthesized nanoparticles. Soot is black, closely resembles a black body radiator, absorbs and emits at all wavelengths, has a complex composition and amorphous structure, and withstands high laser irradiance without significant changes apart from sublimation. Synthesized particles have a chemical composition which is individually rather simple, although covering a wide range on the whole. As a consequence, particular optical properties can be exhibited according to the particle considered. The structure is frequently crystalline, and many physical parameters can change widely from case to case. This leads to a complex frame, easily falling out from the perspectives outlined in Ref. [13], which are nevertheless valid in many cases. Just to make a simple example, while soot incandescence could be excited using a wide range of laser wavelengths, with the choice of the fundamental of the Nd:YAG being dictated by best convenience, the same is not in many cases with particle synthesis. In addition, the reaction of the single species to laser irradiation is nearly unpredictable, as clearly pointed out by Vander Wal et al. in their pioneering work [21].

In this work we present some results on LII experiments on titania nanoparticles produced by flame synthesis in a hybrid burner. Titania is a material of huge interest in many fields and is primarily produced by flame synthesis, as stated above. Then, the possibility of applying LII in titania production is particularly attractive. Preliminary applications have already

been tried, although not in a synthesis flame but on a titania powder fountain [18,19]. In the current stage, the major concern was to investigate the spectral content of the radiation emitted by titania particles under strong laser irradiation, that is the Laser Induced Emission (LIE), in the line of the approach followed in [21], from which the term LIE was also adopted. Both from spectral analysis and from TIRE LIE data it will be shown that LII signals can be obtained under carefully selected experimental conditions avoiding spurious spectral contributions. The technique, when properly exploited, could result in a useful tool for the diagnostic of nanoparticles flame synthesis.

2. Experimental

A sketch of the experimental apparatus is reported in Fig. 1. The description given below is divided in subsections, according to the specific use of the different parts.

2.1. Flame reactor

A hybrid burner similar to the one described by Wooldridge et al. [22] was used to synthesize titania, as well as other oxides. It consists of a porous plug burner (3.6 cm diameter) with a hole drilled in the centre, where a small brass pipe (2 mm outer diameter) is inserted. The first produces a sustaining premixed methane–air lean flame ($\Phi=0.8$), while precursors are injected through the pipe producing an inner, diffusion, reaction flame. The liquid precursor, Titanium Tetraisopropoxide (TTIP, 97% purity Sigma-Aldrich), is stocked into a flask kept at a constant temperature according to the wanted amount to be injected (usually 120 °C). The originated vapors are pushed through the pipe by a dried carrier gas, nitrogen in our case. In such a system the precursor consumption measurement is possible only by weighing at the end of the experimental sessions. By controlling temperature and nitrogen flow, a reproducible, stable flame structure is obtained. The inner part of the burner body including the brass pipe is kept at a constant temperature of 145 °C, while the peripheral part of the burner is water cooled. As a result, a conical premixed flame is obtained, with a thin diffusion flame in the middle, about 80 mm high, flickering in its upper third, with a stable columnar structure below, as it will be described in Section 3.1.

Two kinds of equipment were used to extract powder samples from the flame (not shown in the figure). The first one was a thermophoretic sampler [23] driven by a rapid insertion device, consisting of an electro-pneumatic piston with 10 cm stroke, carrying at its extremity a TEM grid brought in this way in the middle of the flame for a fixed time (50 ms). A second device allowed to suck larger quantities of material through a water-cooled stainless still probe connected with a small pump. The material obtained in this way was used for XRD analysis. Both systems were used to sample the flame all along its height.

2.2. Flame imaging

Pictures of interesting species in the reaction flame have been taken by an intensified CCD Camera (DiCAM-PRO, PCO

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