



Simultaneous measurement of internal and external properties of nanoparticles in flame based on thermophoresis



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ABSTRACT

The mechanism of nanoparticles processed in flame aerosol reactors involves vapor to solid reaction, nucleation, coalescence, agglomeration, diffusion and other processes. Determination of internal (e.g., particle size distribution (PSD), aggregate fractal dimension (AFD), and particle volume fraction (PVF)) and external (e.g., temperature and flow velocity) properties of nanoparticles through numerical simulations or experimental measurements is critical to understanding the underlying particle dynamics, which still remains a major challenge. Multiple key internal and external properties of nanoparticles in flame were measured and characterized simultaneously in this study by a simple and novel dual time-interval thermophoretic sampling (DTTS) method. A tailor-made fine-wire thermocouple was first used to measure flame temperature, with a sufficiently short residence time to reduce the effects of radiation losses and nanoparticles deposition as possible and thereby the thermocouple response met the first-order dynamic equation where only heat convection was considered. Two TEM grids were used for nanoparticle sampling at a position and were exposed to flame for two different time intervals. As the amount of particles deposited on the probe surface by the thermophoretic force is a function of gas temperature, flow velocity, PVF and the probe exposure time in the flame, we proposed an integrative solution for these multiple parameters using the two samples by accounting for the effects of the unsteady temperature gradient of the probe. The effects of flow velocity on convection heat transfer of flame and TEM grids were considered by analyzing the visible microscopic state of thermophoretic-deposited particles. A co-flow diffusion CH_4 flame for TiO_2 nanoparticle synthesis by feeding TiCl_4 vapor was measured via the DTTS method. The experimental measurements of flame temperature, flow velocity and PVF at the different flame heights agree well with the simulation results by coupled computational fluid dynamics with population balance modeling (CFD-PBM).

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1. Introduction

Flame synthesis of nanoparticles is routinely used to make a variety of commercial materials, including TiO_2 , SiO_2 , and Al_2O_3 , amounting to millions of tons annually. They are used industrially as pigments, opacities, catalysts, and for other functions [1,2]. Flame synthesis is a complex process, as all characteristics of the product particles are determined within a few milliseconds and can be influenced by many process variables [3]. Consequently, it is not surprising to find a large number of early research studies focusing on all sorts of tricks and devices to control product characteristics or to facilitate design and operation of flame reactors [4,5]. At present, a clear understanding of flame synthesis of nanoparticles remains a major challenge even though a variety of

scientific communities have studied it closely [3]. The complex chemistry and particle dynamic processes that include combustion, flow and particle evolution need to be explored more deeply by measurement and diagnostic techniques. The concentration distribution and temperature history of reactants (including fuel, oxidants, precursors and particulates) have been shown to be the most important parameters that determine the characteristics of the product particles [6]. In flame reactors, the temperature history is influenced primarily by the flame temperature and gas flow. Therefore, flame temperature, flow velocity, particle concentration (i.e., particle volume fraction (PVF)) and particle internal characteristics (typically particle size distribution (PSD)) must be measured simultaneously.

Flame temperatures as a function of height are typically measured with a fine-wire thermocouple [7]. The measured temperatures are then corrected for radiation loss according to Collis and Williams [8]. It is worth noting that thermocouple measurement

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Nomenclature

a	particle surface area, m^2	T	temperature, K
A_{ai}	projected area of i -th aggregate, m^2	t_e	residence time of TEM grid in flame, s
a_g	gas thermal diffusivity, m^2/s	T_g	actual flame temperature, K
A_i	total area of FSEM image, m^2	T_{sur}	surrounding temperature, K
A_{pi}	projected area of a primary particle within i -th aggregate, m^2	T_{th}	temperature of thermocouple junction, K
C	mole concentration of gas, mol/m^3	T_w	temperature of TEM grid, K
c_g	specific heat capacity of gas, $J/(kg\ K)$	u_g	gas flow velocity, m/s
c_{th}	specific heat capacity of thermocouple junction material, $J/(kg\ K)$	u_T	particle thermophoresis velocity, m/s
c_w	specific heat capacity of TEM grid material, $J/(kg\ K)$	v	particle volume, m^3
D	diameter of thermocouple junction, m	v_a	geometric average volume of aggregate, m^3
d	diameter of lead wire, m	V_p	total volume of deposited particles in a FSEM image, m^3
$d_{a,g}$	geometric mean volume-equivalent diameter of aggregate, m	W_{ai}	projected maximum width of i -th aggregate (normal to maximum length), m
D_f	mass-fractal dimension	W_i	molar mass of the component i
D_L	scaling exponent based on projected area	x	position, distance, m
$d_{p,g}$	geometric mean diameter of primary particle, m		
d_{pi}	primary particle diameter within i -th aggregate, m		
D_T	particle thermophoretic diffusivity, m^2/s		
E_s	activation energy, kJ/mol		
f_v	particle volume fraction		
h	convective heat transfer coefficient, $W/(m^2\ K)$		
k	turbulent kinetic energy, m^2/s^2		
k_B	Boltzmann constant, $1.38065 \times 10^{-23}\ J/K$		
k_f	fractal prefactor		
k_L	prefactor		
Kn	Knudsen number, dimensionless		
L_{ai}	projected maximum length of i -th aggregate, m		
M_p	molecular weight of TiO_2 , $79.865 \times 10^{-3}\ kg/mol$		
n	number density function of particle, $\#/(m^3\ m^3)$		
N_i	number of primary particles within the i -th aggregate		
Nu	Nusselt number, dimensionless		
Pr	Prandtl number, dimensionless		
q_{Cond}	conduction heat flux, W/m^2		
q_{Conv}	convection heat flux, W/m^2		
q_{Rad}	radiation heat flux, W/m^2		
r	reaction rate, $mol/(m^3\ s)$		
R	gas constant, $8.314\ J/(mol\ K)$		
Re	Reynolds number, dimensionless		
S_A	specific surface area, m^2/g		
t	time, s		

Greek symbols

α_{mom}	momentum accommodation coefficient
α_n	normal absorptivity
β	agglomeration rate coefficient (agglomeration kernel), $m^3\ s^{-1}$
δ	thickness of TEM grid, m
ΔT	temperature error, K
ε	dissipation rate of turbulent kinetic energy, m^2/s^3
ε_p	emissivity of TiO_2 particle
ε_{th}	emissivity of thermocouple junction
ε_w	emissivity of TEM grid
θ	dimensionless temperature
ϑ_T	dimensionless particle deposition flux
λ_g	gas thermal conductivity coefficient, $W/(m\ K)$
λ_{th}	thermal conductivity coefficient of junction material, $W/(m\ K)$
ν_g	gas kinematic viscosity, m^2/s
ν_i	stoichiometric coefficient of component i
ρ_g	gas density, kg/m^3
ρ_p	density of TiO_2 particle, kg/m^3
ρ_{th}	density of thermocouple junction material, kg/m^3
ρ_w	density of TEM grid material, kg/m^3
σ	Stefan–Boltzmann constant, $5.67 \times 10^{-8}\ W/(m^2\ K^4)$
τ	time constant, s
τ_s	characteristic sintering time, s

in the presence of the precursor results in the deposition of particles on the wire by thermophoresis and diffusion, which changes the emissivity of the wire and may deteriorate the measurement accuracy. Laser-induced fluorescence (LIF), which has been used in gas phase combustion, was successfully used for flame temperature measurement in dilute particle-laden low pressure flames [9]. Fourier transform infrared (FTIR) spectroscopy is also particularly attractive, as it concurrently provides information on the flame temperature, gas composition and particle concentration during flame synthesis [10]. However, LIF thermometry is hard to carry out and lacks accuracy [11], and FTIR can suffer from isolating the signal region in addition to accuracy [12,13], both of which require novel approaches and further development.

There is not currently an available measurement solution for flow velocity in flame synthesis because most flow velocity measurements depend on micron-sized tracer particles, such as particle image velocimetry (PIV) [14] and laser Doppler velocimetry (LDV) [15]. As is known, the introduction of tracer particles to flame aerosol reactors has deleterious effects on nanoparticle synthesis and the measurement of other parameters, such as PVF. The flow velocity distribution is therefore generally obtained by

numerical simulation, especially for some measurement-difficult object. Many efficient models and algorithms have been developed and applied [16–18]. The accuracy of numerical simulation strongly depends on the models and algorithms used.

The experimental detection and quantitative measurement of particles with sizes below 10 nm are still intractable. Most notably, the characteristic fingerprints of very small particles from molecular spectroscopy are generally not available [19]. A laser-based diagnostic for nanoparticles has recently been developed called laser-induced incandescence (LII) [20]. Complex instruments and data processing algorithms must be used and developed. A few commercial instruments, such as scanning mobility particle sizers (SMPS), are available for measurement of PVF and PSD, based on evaluating their concentration in the gas by sampling analysis [21].

To individually characterize fine particles using electron microscopy (EM) methods, the particles must be collected on special substrates, such as TEM grids, for subsequent detailed analysis by techniques including transmission electron microscopy (TEM), scanning electron microscopy (SEM). Methods of collecting particles onto substrates for microscopy include those employing inertial impaction, gravitational settling, vapor deposition, and

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