EXPERIMENTAL STUDYING DEPOSITION OF ALUMINA SPLATS UNDER COMPLETE CONTROL OF KEY PHYSICAL PARAMETERS

Oleg P. Solonenko, Alexandr A. Mikhalechenko, Andrey V. Shminov and Evgenii V. Kartav
Institute of Theoretical and Applied Mechanics, Siberian Branch of RAS.
4/1 Institutskaya Av., Novosibirsk, RUSSIA

Abstract

Hydrodynamic and thermal physical phenomena associated with impact, spreading out and solidification of melted particle on solid substrate resulting in specific morphology, final thickness and diameter of splat (flattened and solidified droplets) are of exceptional importance for thermal spray and other technologies. The main goal of this paper is studying melted alumina particle—substrate interaction under complete control of the key physical parameters (KPPs) prior to impact—velocity, temperature and size of droplet, temperature of substrate and its surface state.

1. Introduction to experimental setup

To study the interaction of melted particles with a substrate under complete control of the KPPs we singled out a heated particle from a duster plasma jet with the help of a water-cooled diaphragm and subsequently measured its velocity, temperature and sizes. The principal diagram of the setup is shown in Fig. 1. As the particle passes the opening of 1 mm in diameter in the diaphragm D and subsequently traverses the control measurement volume (CMV), the solenoid-driven shutter ES blocks the opening thus separating the particle out of the plasma jet.

To simultaneously measure the velocity, temperature and size of particles, we employed the technique based on light scattering by the particle in combination with two-color pyrometer [1] realized in one CMV. The CMV is located 5 mm away from the water-cooled diaphragm D and forms with the help of an intake lens L₁ (Fig.1, a) and a two-slit diaphragm SD (Fig.1, b), w₁=100 μm, w₂=800 μm, the distance between the slits l=800 μm, and their height of 3 mm). In the two-color pyrometer, the image of the two-slit diaphragm SD is projected onto photocathodes PEM₁ and PEM₂ with the help of the transmitting optics and light splitter LD ahead of which light filters F₁ and F₂ are installed transmitting light in narrow spectral regions around λ₁=0.7212 μm and λ₂=0.8906 μm.

To measure the particle size in the CMV, we used the laser knife of an Ar⁺ laser (λ₁=0.488 μm, output power 1 W) of width 500 μm and height 8 mm formed with the help of a cylindrical lens L₂, spherical lens L₁ and deflecting prism P. In the plane where the image of the intake lens L₁ lays, the laser knife is coincident with the wide slit S₁ (Fig.1, b), and the light scattered by the particle is directed onto the photocathode of PEM₁ with the help of the dichroic mirror DM and the transmitting optics ahead of which the light filter F₁ transparent in a certain spectral region around the laser wavelength. λ₁=0.488 μm is installed. To measure the power emitted by the Ar⁺ laser, the photodiode FD was used, which allowed us to elimi-
nate the detrimental influence of laser instability (the amplitude of low-frequency pulsation of the emitted intensity is within 3 %).

Fig. 1. Schematic of the setup (a) diagnostic complex, (b) multi-slit diaphragm PT plasma torch, D diaphragm, FS solenoid-driven shutter, S substrate, PD photodiode, L1, L2, L3 lenses, P deflecting prism, SD diaphragm, DM dichroic mirror, GF set of neutral light filters, FD light splitter, M mirror, F1, F2, F3 light filters, PEM1, PEM2, PEM3 photoelectronic multipliers, BIS discriminator, ADC analog-to-digital converter.

The signals generated by photo-detectors PEM1, PEM2, and PEM3 are recorded by 4-channel analog-to-digital converter (ADC) [2]. The maximal frequency of signal discretization is 40 MHz. Amplified signal from PEM3 enters the discriminator BIS (the channel of scattered light detection) that generates standard signal of ADC start if the level of scattered signal is higher than that of the threshold. The discriminator is applied to prevent false start caused by background radiation.

The substrate S is placed 4 mm away from the CMV. It is rigidly fixed to the surface of an ohmic heater equipped with a thermocouple, which allows us to vary the substrate temperature up to 600 K and measure it.

2. Experimental procedure, results and discussion

To measure the velocity, temperature and size of a singled out particle prior to its collision with the substrate, we use both methods based on registering the thermal radiation emitted by the particle and a combination of laser-optical methods permitting independent determination of the particle size from the absolute intensity of scattered light. This allows us to analyze the pyrometric signal at one wavelength and independently determine the temperature of the particle with allowance for its emissive characteristics. Typical oscillograms of the pyrometric signals and that proportional to the intensity of scattered light are shown in Fig. 2. Amplitude $N_0$ of the signals is expressed in digits, the sample time is the unit of axis of abscissae, $N_0$ is a number of time samples.

The time-of-flight technique for luminous particles in combination with pyrometer is realized for a chosen geometry of the multi-slit diaphragm SD (Fig. 1, b) allows us to simultaneously put into correspondence to each particle certain values of its axial velocity and surface temperature [2].

2668
Parameters of particle are being found with the help of correlation analysis of real and model pyrometric signals in combination with estimating by least-squares method.

Fig. 2. The characteristic views of pyrometric signals and scattered one.

For each spectral region, the model signal is computed according to the formula

\[ I_i(t) = \sum \phi_i(\lambda_i) \delta(D_{i,\lambda}, \varphi_i, v_p, \phi_i, T) \int \frac{d\lambda}{\lambda} \frac{C_i d\lambda}{\lambda^2 \exp(C_i / \lambda T)} \]

where \( I_i(D_{i,\lambda}, \varphi_i, v_p, \phi_i, T) \) is the function that describes passing of the image of a spherical particle of diameter \( D_{i,\lambda} \) in the plane of the intake diaphragm \( S_i \) in time as a function of its velocity \( v_p \). This function is proportional to the area of the particle image sequentially cut by slits \( S_i \) (Fig. 1, b) of the intake diaphragm; \( \alpha \lambda, T_i \), \( i=1, 2 \) is the particle emissive power at the center of the spectral region of the \( i \)-th interference filter with the measured transmission factor \( \Phi_i(\lambda) \), \( \delta(\lambda_i-\lambda, \lambda_i-\lambda_i/2) \) is the transmission factor of the \( i \)-th optico-electronic path determined preliminarily and relating the radiation fluxes emitting by the particle to the registered values of the analog signals. The algorithm of processing the experimental signals (realization) corresponding to single out particle is the following. First, the axial velocity \( v_p \) is to be found by maximization of the cross-correlation function between recorded physical realization \( R_i(t) \), \( i=1, N_2 \) and the realization \( R_{\text{mod}}(t) \), \( i=1, N_3 \) computed for model (test) particle, where \( N_3 \) is the length of the buffer memory reserved for each channel in ADC for data acquisition. To reduce the time required for processing experimental data, a priori information is used about ranges in which the size, velocity and temperature of the particle could vary. With the known particle velocity, one can find the position of the constant component (under actual conditions of a plateau with a noise) of trapezoidal pulses in two working spectral regions and determine the color temperature of the particle \( T_i \) from the relation (2) (gray body approximation)

\[ F(\lambda, \lambda, T, T_i) \]

where

\[ \int_{\lambda_{i-1/2}}^{\lambda_{i+1/2}} \Phi_i(\lambda) \frac{C_i d\lambda}{\lambda^2 \exp(C_i / \lambda T_i) - 1} \]

and

\[ \frac{U_i}{U_i \exp(C_i / \lambda T_i) - 1} \]
where \( I_1 \) and \( I_2 \) are the average values of the amplitudes of the trapezoidal signals in the spectral regions of interest.

The particle size \( D_p \) is to be found with the help of the least-squares method applied both to the experimental realization and the model one for the found values of \( \gamma_p \) and \( T_p \) at \( \lambda_p = 721 \, \mu m \) in the region of the constant component of the trapezoidal signal with allowance for the emissive power of the particle. To determine the temperature and size of particle, the coefficients \( \xi(\lambda_p) \) and \( \delta(\lambda_p) \) were preliminarily measured in calibration tests.

The emissive power is estimated by analyzing the absorption characteristics of a spherical particle according to the Kirchhoff law [3], i.e. \( e(\lambda, T) = Q_{em}(\lambda, T) \), where \( Q_{em}(\lambda, T) \) is the absorption efficiency factor. It is known that the absorption efficiency factor \( Q_{em}(\lambda) \) of spherical particle depends on the complex refractive index of particle material \( \xi \) and on the scattering parameter \( \eta \). The alumina whose optical properties are most accurately described in visible-spectral band for wide temperature variety has been chosen as base material, with approximation formulas being mentioned in [4, 5].

To analyze the spectral dependence \( Q_{em}(\lambda) \) of spherical alumina particle one can use the approximation formula reported in [4] as well as spectral dependencies for \( \nu = 3 \mu m, \xi = 2 \mu m, \eta = 2 \mu m \) in [5]. Since the analysis of radiation of alumina particles in the vicinity of melting temperature (liquid – solid phase) \( T \approx [2200; 2400] \, K \) fulfilled in [6] shows that there is a violation of Kirchhoff law. Therefore the estimate of temperature based on thermal radiation of particle becomes ambiguous, though being correct at least for liquid phase.

Regardless of measuring the size of singled out particles, we also use the light-scattering technique. The measured intensity of the scattered light is related to the true intensity of the scattered radiation by the formula \( I_{s,0} = \xi(\lambda_p) P_{s,0} \), where \( \xi(\lambda_p) \) is the transmission factor of the optical-electronic channel and \( P_{s,0} \) is the total power of the light scattered by a particle at the axis of the laser beam, determined by the intensity of the scattered light \( I_{s,0}(r, \theta, \phi) \) and the solid angle of the intake lens \( L_1 \) (Fig. 1, a).

\[
P_{s,0} = \int_{2\pi} I_{s,0}(r, \theta, \phi) \, d\phi
\]

The intensity of the scattered radiation in the far zone is given by the following formula

\[
\lim_{r \to \infty} I_{s,0}(r, \theta, \phi) = \frac{1}{2\pi} \frac{1}{\mu_0 c} \left[ |S_1(\theta, \phi)|^2 + |S_2(\theta, \phi)|^2 \right]
\]

where \( \mu_0 \) is the magnetic permeability of free space and the velocity of light in it, \( \lambda \) is the wave number, and \( S_1(\theta, \phi) \) and \( S_2(\theta, \phi) \) are elements of the scattering matrix [3].

If the center of a spherical particle is situated at the axis of the laser beam (axial scattering by a moderately focused beam \( w_b \gg \lambda \) (\( w_b \) is the half-width of the incident beam), then the solution becomes almost as simple as the one for the case of plane-wave scattering [7].

Using available data on optical properties of alumina (refractive index \( n(\lambda, T) \) and absorption factor \( \nu(\lambda, T) \) ), we calculated the angular distribution of the function \( I_{s,0}(r, \theta, \phi) \) for spherical \( Al_2O_3 \) particles situated at the beam axis and scattering a focused laser-emitted light in the temperature range 1700-3000 K. In Fig. 4 there are drawn curves of dependence of light power \( P_{s,0} \) scattered by \( Al_2O_3 \) particles on size of particle in accordance with (3) at different temperatures of particle in the case being close to that of back-scattering \( \theta = 140^\circ \), \( Al2 = 8.5^\circ \), \( \phi = 90^\circ \), \( w_b = 8.5^\circ \).

As seen from the shown dependencies that in fact amount of scattered light for geometry chosen doesn’t depend on temperature of particle, whereas the uncertainty of size determina-
tion at various temperatures of particle doesn’t exceed that concerning oscillations of scattered power at room temperature, namely it is within ±3 μm.

To determine the calibration factor $\xi(\lambda)$ for the particle size meter, we used a weakly dusted flow (at room temperature) of preliminarily spheroidized $\text{Al}_2\text{O}_3$-particles with sizes $D_p$ ranging between 20 and 60 μm. The experimental procedure included experiments on light scattering in the regime of registering light from singled out particles, deposition of these particles onto a substrate wetted with glycerin, and measurement of the particle size with the help of a microscope. Figure 5 shows results of measurements of the intensity of the scattered light for various particle sizes, the theoretically predicted dependence, and the obtained value of the calibration factor. The calibration procedure for the two-color pyrometer was described at sufficient length in [2], and calibration results are shown in Fig. 3.

![Fig.4. The curve of scattered intensity computed theoretically as function of size of particle at different temperatures of $\text{Al}_2\text{O}_3$-particles ($\theta=140^\circ$, $\lambda=8.5$, $\phi=90^\circ$, $D_p=8.5$).](image)

![Fig.5. Theoretical dependence of scattered light power on size of particles (solid line) and experimental data](image)

**Table 1. Experimentally determined characteristics of $\text{Al}_2\text{O}_3$-particles**

<table>
<thead>
<tr>
<th>No</th>
<th>$T_{\text{meq}}$</th>
<th>$v_{\text{rel}}$</th>
<th>$N$</th>
<th>$T_e$, K</th>
<th>$R_{\text{p, cm}}$, μm</th>
<th>$R_{\text{e, cm}}$, μm</th>
<th>$T_e$, K</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>212</td>
<td>92.0</td>
<td>80</td>
<td>1885±150</td>
<td>55.4</td>
<td>30.4</td>
<td>2150.</td>
</tr>
<tr>
<td>52</td>
<td>212</td>
<td>116.0</td>
<td>80</td>
<td>2058±165</td>
<td>42.8</td>
<td>-</td>
<td>25.3</td>
</tr>
<tr>
<td>53</td>
<td>208</td>
<td>108.0</td>
<td>80</td>
<td>2060±165</td>
<td>35.8</td>
<td>-</td>
<td>22.9</td>
</tr>
<tr>
<td>59</td>
<td>218</td>
<td>134.7</td>
<td>70</td>
<td>2691±215</td>
<td>20.4</td>
<td>24.30</td>
<td>30.2</td>
</tr>
<tr>
<td>60</td>
<td>201</td>
<td>92.7</td>
<td>144</td>
<td>2760±166</td>
<td>13.4</td>
<td>24.65</td>
<td>20.4</td>
</tr>
<tr>
<td>61</td>
<td>110</td>
<td>148.7</td>
<td>100</td>
<td>2900±203</td>
<td>9.2</td>
<td>25.68</td>
<td>14.8</td>
</tr>
<tr>
<td>65</td>
<td>107</td>
<td>158.0</td>
<td>84</td>
<td>2797±224</td>
<td>17.6</td>
<td>25.21</td>
<td>26.0</td>
</tr>
</tbody>
</table>

Table 1 exemplifies measurement results for the following parameters of alumina particles: velocity $v_{\text{rel}}$, $N$ - estimated number of counts on the plateau of trapezoidal signals; color temperature $T_e$; error of its measurements caused by the noise contained in pyrometric signals; particle size calculated from the amplitude of the pyrometric signal for the particle radius $R_p$; calculated from the amplitude of the pyrometric signal at known color temperature $T_e$, with allowance for the emissive power; particle temperature $T_{\text{e, cm}}$, estimated with allowance for the fact that particles in reality are not perfectly gray bodies and for the value of the particle size.
$R_\text{sc}$ recalculated for the obtained temperature; particle size $R_\text{ps}$ measured from the intensity of the scattered light; particle temperature $T$ calculated from the amplitude of the pyrometric signal with allowance for the size and emissive power of the particle.

The analysis performed permits estimation of the confidence level of the obtained particle characteristics in plasma jet. In Fig. 6 there are shown the photos obtained using SEM method, with numbers being in concordance with those depicted in Table 1.

![Fig. 6. The photos of aluminum splats obtained using the SEM method with numbers being in concordance with those of substrates.]

3. Conclusions

The particle size determined by magnitude of pyrometric signal for both color temperature and correction of real non-gray character of radiation is substantially affected by accuracy of temperature estimate. It results in systematic overestimation of particle size. Probably, this fact is caused by valuable temperature gradient inside of particle, as either radiation of surface layer and bulk radiation transfer of particle form thermal radiation of particle itself. The application of laser-optical method of size estimation weakly dependent on temperature based on absolute intensity of scattered light allows one to perform cross-testing of the methods and estimate a correlation of obtained results as well.

Acknowledgement
This research was supported in part by Russian Foundation for Basic Research (Grant No 98-02-17810) and by Siberian Branch of the Russian Academy of Sciences in the framework of the Interdisciplinary Program (Project No 45).

References

2672