Optical emission generated by particle impact during aerosol deposition of alumina films

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ABSTRACT

Aerosol deposition (AD) is a ceramic coating process that enables films to be fabricated by microparticle impact at room temperature. The optical emission during AD reported in previous studies is thought to be fracto-emission or triboemission; however, the mechanism has never been investigated in detail. In this study, the optical emission of the electric discharge generated during aerosol deposition of alumina films using He, Ar, and N\(_2\) was measured by optical emission spectroscopy. Each spectrum had peaks corresponding to the emission peaks of the carrier gas and no blackbody radiation characteristics were observed. The emission intensity was proportional to the flux of the kinetic energy of the aerosol particles, which suggested that the emission occurred via fracto-emission during particle impact. The gas temperature remained low at around 300 K, which quantitatively confirmed that AD is a room-temperature process.

1. Introduction

Aerosol deposition (AD) is a ceramic coating process that enables films to be fabricated by microparticle impact at room temperature [1,2]. In AD, ceramic or metal microparticles 0.1–10 \(\mu\)m in diameter that are accelerated to 100–500 m s\(^{-1}\) by a gas flow collide with a substrate, resulting in the formation of a dense film. AD has been used for applications such as plasma-resistant coatings [3], microactuators [4], solid oxide fuel cells [5], lithium ion batteries [6], hydrophobic coatings [7], and flexible electronics [8]. The film formation mechanisms of AD are still under debate. One possible mechanism is room-temperature impact consolidation, which involves plastic deformation of the ceramic microparticles [9]. This deformation behavior was observed by microcompression tests on nanopillars [10,11] and nanometer- or micrometer-sized particles [12–15]. Molecular dynamics calculations suggest that the microparticles accelerated in AD processes may show plastic deformation [16–19].

Optical emission during AD is a unique phenomenon that is not well understood [20,21]. Optical emission and electric discharge during AD of Pbi(Zr,Ti)O\(_3\) was observed when He gas was used, although no emission was observed when N\(_2\), O\(_2\), or air was used [20]. Because only the films formed using He were black it was suggested that the electric discharge affected the film properties. Fracto-emission or triboemission was proposed as the generation mechanism of the optical emission [20,21]; however, details of the optical emission have never been reported. In the densification
mechanism of AD, it is necessary to clarify whether the luminescence is accompanied by heat generation. Triboeission and fracto-emission are sometimes accompanied by heat generation. In a triboeission experiment where SiO$_2$ glasses and crystalline quartz were cut with a rotating diamond-impregnated saw blade, the blackbody emission spectra were recorded and the temperature was estimated to be around 2000 K [22]. The photoemission spectra from rock fracturing has also been characterized by blackbody radiation, and the temperature was estimated to be 1750 K [23]. Therefore, the particle impact during AD could generate large amounts of heat and the deposition may occur via particle melting or high-temperature plastic deformation. It has been proposed that melting of the particle surface is crucial in bonding, as observed in shock compaction [17]. To determine whether this is the case, the mechanism by which the optical emissions during AD are generated must be clarified.

Understanding this mechanism also has interdisciplinary value because similar luminescence has been observed in other fields. Electrified dust clouds can cause electrical breakdown, as observed in volcanic lighting [24,25]. Spark discharges are characteristic of volcanic lightning because the dust clouds are in atmospheric-pressure air, in which glow discharges rarely occur. Glow-like discharges are induced by dust motion at low pressures, as observed in experiments simulating the surface of Mars [26–29]. These discharges are expected to be closely related to triboelectric charging [30], fracto-emission [31], triboeission [32], and exo-electron emission [33]. Furthermore, the electric discharges in AD processes contain a considerable number of fine particles, which may result in conditions similar to dusty (complex) plasmas, which have been extensively studied in thermodynamics and statistical mechanics [34].

During AD processes, the kinetic energy of a particle on impact is partly converted into its strain, fracture, and bonding energies, which results in the formation of a film. This energy conversion also involves energy dissipation, such as heat generation and discharge generation, at least in certain cases [20,21]. The sintering mechanism of AD appears to be different from conventional sintering; however, considering the optical emission generated during the AD processes, the deposition could be caused by the heat generated by the particle impact. To determine whether this is the case, it is necessary to clarify the mechanisms of the optical emission that occurs during AD. In this study, we examined the discharges during AD by optical emission spectroscopy.

2. Experimental

The schematic of the experimental setup is shown in Figure 1. The deposition system was the same as in previous reports [1,4]. The main chamber was evacuated by a mechanical booster pump and a rotary pump, whose base pressure was 40 Pa. In a mechanically vibrated aerosol chamber, single-crystal α-alumina microparticles (average diameter 0.38 µm, AA-03, Sumitomo Chemical Co., Ltd.) were dispersed as an aerosol by a gas flow (He, N$_2$, and Ar; flow rate: 1–20 standard liter per minute [SLM]) and accelerated toward a borosilicate glass substrate in the main chamber by a nozzle (orifice of 0.4 × 10 mm). The alumina powder consisted of single crystals grown by chemical vapor deposition and showed a low AD deposition efficiency [35]. However, this powder had a narrow size distribution with a single peak, making it suitable for spectroscopic analysis. The pressures inside the main chamber increased to 2000 Pa with the gas flow, according to the flow rate. The gap between the nozzle and the substrate was 5 mm during spectroscopic measurements. The gap was widened to 20 mm while the photographs were taken to reveal the spatial distribution of the optical emission. These experimental conditions are typical of film fabrication with AD [1,2]. The light emitted from the gap was collected by optical lenses through the SiO$_2$ glass window and analyzed by a spectrometer (Glacier X, B&W TEK Inc.). The spectrum of the N$_2$ second positive system (2PS), emitted by the transitions from the C $^3\Pi_u$ state to the B $^3\Pi_g$ state, was analyzed following the literature [36].

3. Results and Discussion

3.1. Optical emission during AD using He, Ar, and N$_2$

Figure 2(a) is a photograph of the nozzle and a substrate. The aerosol flow goes up through the nozzle and collides with the substrate. Figure 2(b-d)
**Figure 2.** Photographs of the nozzle and the substrate with a gap of (a) 5 mm and (e) 20 mm. Photographs of the discharge with a gap of 5 mm using (b) He, (c) Ar, and (d) N$_2$ with a flow rate of 10 SLM. Photographs of the discharge with a gap of 20 mm using (f) He, (g) Ar, and (h) N$_2$ with a flow rate of 15 SLM. Aerosol flow ascends through the nozzle and collides with the substrate. The light visible above the substrates is due to the reflection from the substrates.

**Figure 3.** Optical emission spectra for He, Ar, and N$_2$ carrier gases with a flow rate of 15 SLM. The peaks marked with an asterisk in the He spectrum are from the N$_2$ impurity.
are photographs of the discharges generated during AD using He, Ar, and N₂, respectively, with a gap between the nozzle and substrate of 5 mm. Glow-like discharges that spread between the nozzle and the substrate were generated when He was used, whereas strong optical emissions near the substrates that spread horizontally were observed when Ar and N₂ were used. Figure 2(e-h) are photographs of the discharges when the gap between the substrate and the nozzle was 20 mm. Similar to the discharges observed with a gap of 5 mm, the optical emission spread between the nozzle and the substrate when He was used. The optical emission was most intense near the substrate for all gases. Shock waves were formed owing to the supersonic flow, as suggested by numerical simulations [37,38].

Figure 3 shows the optical emission spectra of the discharges generated when He, Ar, and N₂ were used with a flow rate of 15 SLM. Each spectrum had peaks corresponding to the emission peaks of the carrier gas. There was no broad and continuous spectrum as seen in blackbody radiation. For He, strong emissions from the N₂⁺ first negative system [36] were observed, which originated from impurity N₂ (asterisks in Figure 2). The trace N₂ gas shows considerable emission intensity because metastable He (He*) ionizes and excites N₂ efficiently via the following reaction [39,40].

\[ \text{He}^* + \text{N}_2 \rightarrow \text{N}_2^+ + \text{He} + e^- \]

In contrast, no emissions were observed when Ar and N₂ were used in previous work [20], which may be because of differences in the aerosol particles.

### 3.2. Dependence of emission intensity on flow rate for He

Figure 4 shows the optical emission spectra for He with varying flow rate. The emission intensities increased with flow rate. Figure 5(a) shows the dependence of emission intensity of each peak on flow rate for He. Further analysis was conducted considering the aerosol properties. The flux of the particles in the aerosol flow can be calculated from the mass of the powder consumed during the experiments, the duration of which was kept as 75 s for the intensity measurements. It has been confirmed experimentally that particle impact velocity increases with the flow rate [1,41]. We assumed that the particle velocity is proportional to the flow rate. A recent numerical simulation has suggested that the impact velocities of Al₂O₃ particles with a size smaller than 1.0 μm are proportional to the flow rate, which supports our assumption [42]. The flux of the kinetic energy of the particles in aerosol flow, \( I_k \), was expressed as

\[ I_k = C \times I_p \times F^2 \]

where \( C \) is a constant, \( I_p \) is the flux of the particles in the aerosol flow, and \( F \) is the gas flow rate. Figure 5(b) shows the relationship between the peak intensities and \( I_p \times F^2 \) for three emission peaks. The peak intensities were proportional to \( I_p \times F^2 \) (gray lines), which suggested that the optical emission was increased by the particle impact.

### 3.3. Rotational and vibrational temperatures of N₂

Figure 6 shows the dependence of rotational temperature and vibrational temperature of N₂ calculated from 2PS on the flow rate for N₂ carrier gas. The gas temperature can be estimated from the rotational temperature, which approximately equilibrates with the gas temperature [36,43]. The measured rotational temperatures were around 300 K, irrespective of flow rate, indicating that the discharges did not generate much heat. The vibrational temperature was much higher, at around 3000 K. Thus, the optical emission measurements suggested that in the discharges, the temperature of the electrons, which excite gas molecules, was much higher than that of the surrounding gas. Although further research is required to understand the slight decrease in vibrational temperature with the increase in flow rate, it could be caused by the shorter duration of gas with a higher flow rate or by the electron temperature change in the plasma [44]. The increase in flow rate increases the pressure inside the chamber and aerosol density, which strongly affects discharge properties. In particular, introducing microparticles into a plasma decreases the electron density and increases the electron temperature [45].

### 3.4. Temperature increase during particle impact

A proposed film formation mechanism for AD processes is room-temperature impact consolidation [9]. It is suspected that the temperature increases during particle impact and that deposition occurs by particle melting. In addition, the previous finite element method simulation, which calculated the local temperature and pressure of an α-alumina particle with a size of 0.3 μm and an impact velocity of 300 m s⁻¹, suggested that the maximum temperature is around 550 K in the small contact area between the particle and the substrate [1]. Another finite element method calculation suggested that the maximum temperature was around 1250 K in a small part (a mesh with a size of 12.5² nm²) of the contact area when two alumina particles with a diameter of 0.5 μm successively hit the alumina substrate at an impact velocity of 350 m s⁻¹ [17]. A recent molecular dynamics simulation suggested that the maximum temperature of a TiO₂...
particle with a size of 0.3 μm was 600 K at an impact velocity of 300 m s⁻¹ and 1000 K at an impact velocity of 400 m s⁻¹ [18]. Because the melting points of Al₂O₃ and TiO₂ are higher than 1800°C [46,47], these calculations suggest that little particle melting is caused by particle impact. Although the sintering temperatures are lower than the melting temperatures [48,49], they are still much higher than the calculated temperatures.

The calculated temperatures depend on the calculation method and calculation conditions. Despite the many simulations, in situ temperature measurements during AD processes have never been reported. This study shows that the temperature of the gas near the impacting particles, where the optical emissions originated, remained around room temperature during the processes, and no black-body radiation characteristics

Figure 4. Optical emission spectra for He with varying flow rate. The peaks marked with an asterisk in the He spectrum are from the N₂ impurity.
were observed. If particles with a temperature over 1000 °C stay on the substrate or bounce off and disperse, a broad spectrum originating from blackbody radiation could be measured.

These results suggest that AD films are formed at low temperatures without particle melting. This study quantitatively confirmed that AD is a room-temperature process. Thus, the densification mechanism must be different from conventional sintering or high-temperature plastic deformation.

3.5. Generation mechanism of optical emission during particle impact
The generation of the discharges in this study may be related to fracto-emission, as suggested in the literature [20,21]. Fracto-emission is the emission of electrons, ions, and photons during fracture. The proposed mechanism is that discharges are generated by the strong electric fields caused by charge separation during fracture [50]. In our work, the alumina particles were fractured by particle impact on the substrate,
which could result in fracto-emission (Figure 7). Electron emission from alumina via fracto-emission has been reported [51]. This idea is supported by our observation of the relationship between emission intensities and the flux of the kinetic energy of the particles. Considering that the kinetic energy $K$ was converted to the energy of the newly formed surface $y \times A$ ($y$: surface energy density, $A$: area of the newly formed surface) at a constant rate $C'$, the following equation holds,

$$yA = C'K$$

Given that $y$ is constant, our estimated kinetic energy is proportional to the area of the newly formed surface. The increase in the surface area of the fractured particles should increase the discharge probability and the optical emission intensity. Furthermore, the optical emission was most intense near the substrate (Figure 1), which also supports this mechanism. However, the discharges spread in the gap and out of the gap, which could be explained by fracto-emission from impacts between incident particles and particles scattered by the substrate.

Previous studies on fracto-emission and triboemission have reported blackbody radiation and large temperature increases up to 2000 K [22,23]. Fracto-emission and triboemission are not necessarily accompanied by heat generation, which only occurs when there is intense friction. In particular, with respect to electron emission, the emission originating from heat generation is distinguished as thermally stimulated exoelectron emission [52]. The optical emission during AD processes is a type of fracto-emission that does not involve heat generation.

In astronautics, a similar type of particle impact is called hypervelocity impact, which simulates the collision between a spacecraft and space debris or meteoroids [53]. Hypervelocity impact with velocities on the order of tens of kilometers per second generates plasmas [53]. These plasmas are generated by the vaporization and ionization of the target substrates driven by shockwaves [54]. In our system, no optical emission from the substrate was observed, and the gas temperatures remained around room temperature. Furthermore, the particle velocity has been reported to be on the order of $10^3$ m s$^{-1}$ [41], which is much lower than that of hypervelocity impact experiments. Therefore, the generation mechanism of the discharges in this study was different from that of hypervelocity impact. The discharges and optical emission associated with the particle motion have been observed in various phenomena, such as volcanic lightning [24,25]. Our simple, continuous generation of particle impact discharge may provide a useful experimental platform for understanding this type of discharge.

Figure 7. Proposed mechanism for the optical emissions observed during AD.
4. Conclusion

We studied the optical emission of the discharges during AD processes. The optical emission was observed when He, Ar, and N₂ carrier gases were used. Each spectrum had peaks corresponding to the emission peaks of the carrier gas. The emission intensities were proportional to the kinetic energy of the aerosol particles, which suggested that the emission occurred via fracto-emission during particle impact. The gas temperatures estimated from the optical emission spectra of N₂ remained around 300 K and no characteristic blackbody radiation was observed. These results supported the idea that AD processes do not involve particle melting or high-temperature plasticity. This study quantitatively confirmed that AD is a room-temperature process.

Disclosure statement

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