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Signature of nano alumina condensation during metal combustion

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ABSTRACT

The work is focused on thermal radiation originating from aluminum oxide nanoparticles formed during the combustion of an aluminum and copper oxide (Al/CuO) thermite. The light emission signature allows for the identification of condensation features important for the global energy balance of burning metal-containing systems. From a fundamental science perspective, identifying light emission signatures associated with phase changes, and in particular, with condensation that is accompanied by high energy release, will lead to new knowledge with widespread applications. The recently developed calibration-free pyrometry approach is used to isolate the radiation of interest during combustion. Processing the obtained spectra made it possible to detect the solidification of the resulting nano alumina. Knowledge of the phase transition temperature enabled the determination of the temporal behavior of the absolute temperature of aluminum oxide nanoparticles during their formation by condensation from gaseous metal suboxides. This ability to measure absolute temperature, as opposed to its reciprocal value available with the original calibration-free pyrometry approach, further enhances the diagnostic method's capabilities. The general implications of revealed peculiarities of nano alumina formation for detailed modeling of the combustion of metal-containing systems are discussed.

1. Introduction

The combustion of metals is a research focus for the energetic materials community due to their high energy densities [1,2]. It is traditionally believed that the release of energy during metal combustion is determined by the burning time of the metal particles [3,4]. Although this statement is generally not incorrect, it is too crude to describe the process at the level necessary for accurate modeling. The possibility of different combustion regimes of the metal particles [5,6], and, consequently, different burning times corresponding to these regimes, makes the burn time a characteristic that is not clearly determined experimentally. Then, the burn time may not accurately describe the rate of energy release. A more serious issue with the "burn time" approach to modeling energy deposition is that this concept ignores the details of the energy release mechanism. Modeling energy conversion requires nuances of the energy release mechanism to be resolved. The implications of oversimplifying the energy release mechanism have been discussed in our recent work [7] and references therein.

In short, most metal combustion energy is released during condensation of gaseous metal suboxides. For example, in the case of aluminum (Al) combustion, condensation energy can account for about 100 % of the total energy of metal combustion [8]. In its turn, the peculiarities of the condensation process at the atomic level are associated with the value of the condensation energy, which is on the order of 5 eV (or more) per condensing molecule [9]. There are semi-empirical descriptions of condensation that consider the above-mentioned features of energy release [10,11]. However, the lack of rigorous concepts on the condensation of gaseous metal suboxides does not allow accurate modeling of metal combustion. Modeling condensation, i.e., the process responsible for most of the energy release, remains the weakest point [12].

Condensation of gaseous metal suboxides in flames, which results in formation of nano-oxides, follows reactions of metals with oxidizers. The co-occurrence of these two processes, that is, the oxidation of metal particles and the generation of nano-oxides, makes it difficult to isolate the processes' signatures while studying the system. In particular, their co-occurrence is the reason for the limited information on the details of the growth of nano-oxides that are essential for describing condensation processes.

In this work, we study the light emission during combustion of an aluminum and copper oxide (Al/CuO) thermite. Due to its calibration-free nature, and therefore, its high resolution, the recently established

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data processing approach [13] enables isolating the thermal radiation originating from alumina nanoparticles generated in the system. Using this approach, we were able to identify the onset of condensation and describe the temporal evolution of formed nano-oxides including their temperature history. In addition, the detected solidification of nano alumina was utilized to infer the absolute temperatures, as opposed to reciprocal values available with the original calibration-free pyrometry approach [13]. The manuscript also describes how changes in emissivities due to phase transitions can be incorporated into calibration-free pyrometry.

2. Phase change indication in calibration-free pyrometry

Calibration-free pyrometry [13] is able to validate assumptions applied for the interpretation of light emission, and, therefore, allows one to distinguish between the single-temperature and multi-temperature nature of the radiation in reactive systems. In this Section, we expand the capabilities of the method and demonstrate how it can be used to identify a phase transition in a system emitting thermal radiation.

Following Ref. [13], the signal from a photomultiplier tube (PMT), which detects light emission, can be expressed by Eq. (1).

$$PMT = \frac{K\varepsilon_{\lambda}}{\exp\left(\frac{C}{\lambda T}\right) - 1} \approx K\varepsilon_{\lambda}\exp\left(-\frac{C}{\lambda T}\right)$$
(1)

Here, $C = 14,388 \ \mu m$ -K is the second radiation constant, λ is the wavelength of a PMT bandpass filter, *T* is the temperature of the emitting body. The second expression in the right-hand side (RHS) of Eq. (1) is deduced from the Wien's approximation of the Planck's law in the visible light that is valid at typical combustion temperatures. Note that the factor λ^{-5} that enters the Planck's function is included in the PMT-dependent constant *K*. Unlike Ref. 13, Eq. (1) explicitly shows the wavelength-dependent emissivity of the radiating body, ε_{λ} that entered the PMT-dependent constant, *K*, in the original work.

The reciprocal temperature can be obtained from signals measured by a pair of PMTs at wavelengths λ_1 and λ_2 , as shown in Eq. (2).

$$\frac{1}{T} = \frac{\ln\frac{K_1}{K_2} + \ln\frac{\varepsilon_{\lambda_1}}{\epsilon_{\lambda_2}} - \ln\frac{PMT_1}{PMT_2}}{\frac{C}{\lambda_1} - \frac{C}{\lambda_2}} \equiv A_{12} - B_{12}\ln\frac{PMT_1}{PMT_2} + B_{12}\ln\frac{\varepsilon_{\lambda_1}}{\varepsilon_{\lambda_2}}$$
(2)

In Eq. (2), constants A_{12} and B_{12} are characteristics of a given pair of PMTs. The normalization constant B_{12} can be exactly calculated according to its definition by Eq. (3).

$$B_{12} \equiv \frac{1}{\frac{C}{\lambda_1} - \frac{C}{\lambda_2}} \tag{3}$$

The third term in the RHS of Eq. (2) is determined by the electrical properties of the emitting body and can be assumed constant in the absence of phase transitions. Then, it was included in the offset constant, A_{12} , in the original work introducing the calibration-free data processing approach [13]. During a phase transition from phase α to phase β , this term experiences a jump, $D_{\alpha-\beta,12}$, described by Eq. (4), in which the subscripts α and β denote the values corresponding to these phases at the given wavelengths λ_1 and λ_2 .

$$D_{\alpha-\beta,12} \equiv B_{12} \left[\left(\ln \frac{\varepsilon_{\lambda_1}}{\varepsilon_{\lambda_2}} \right)_{\beta} - \left(\ln \frac{\varepsilon_{\lambda_1}}{\varepsilon_{\lambda_2}} \right)_{\alpha} \right]$$
(4)

In the general case, the magnitude of this jump depends on the choice of the combination of wavelengths, so, for example, $D_{\alpha \cdot \beta, 12} \neq D_{\alpha \cdot \beta, 13}$. Then, if the time-dependent plots of $B_{ij} \ln \frac{PMT_i}{PMT_j}$ which characterize the temporal behavior of the reciprocal temperature [13], demonstrate a reliable jump in the offset constant, the jump is an indication of a phase transition. Based on the emitting material properties, the temperature of the corresponding phase transition can be considered known. The

known phase transition temperature can further be used to determine the absolute temperature (i.e., transition from the reciprocal temperature to its absolute value) as described by Eq. (6) in Ref. [13] with T_0 being the phase transition temperature.

There are two features of the third term in the RHS of Eq. (2), that are important for analysis, namely, 1) the independence of the term on temperature, and 2) a jump during a phase change. These two features are based on the mechanism of light absorption in condensed matter. The intensity of the process depends on the concentration of electrons in the conduction band, and the spectral behavior is determined by transport properties. In wide-bandgap dielectrics such as aluminum oxide, the electron concentration in the conduction band is strongly dependent on temperature, leading to the well-known increase in the imaginary part of the refractive index of alumina with temperature [14]. At the same time, since the temperature dependence of the imaginary part of the refractive index is decoupled from its spectral dependence [14], the ratio of the imaginary parts of the refractive index at different wavelengths does not depend on temperature. As a result, the ratio of absorptivities/emissivities, which depends on this ratio of the imaginary parts of the refractive index, can also be considered independent of temperature. The natural change in the transport properties of the material during a phase transition affects the spectral behavior of the imaginary part of the refractive index, resulting in a jump in its ratio at given wavelengths, and consequently, in a jump of the ratio of emissivities.

3. Experimental

The experiment was designed to observe the condensation of gaseous aluminum suboxides that results in the formation of alumina nanoparticles. An Al/CuO mixture was used in the study. When in contact with alumina produced by thermite combustion, aluminum can react to generate gaseous alumina suboxides [6], which subsequently condense. The main focus of data analysis was on discovering this process.

The powder burning set-up is described elsewhere [13] and sketched in Fig. 1. A 50 mg stoichiometric powder mixture that consisted of aluminum powder (Product Number US1043, US-Research Nanomaterials, Houston, TX, USA) and copper (II) oxide powder (Product Number 208841, MilliporeSigma, Burlington, MA, USA) was placed in the chamber surrounded by an air environment. The powder was irradiated by a 40-Watt CO₂ laser (10 μ m wavelength band, Firestar TI-Series, Synrad, Mukilteo, WA, USA) that initiated thermite reaction. A Phantom v2512 high-speed camera (Vision Research, Wayne, NJ, USA) recorded the reaction event at the nominal rate of 10,000 frames per second.

Light emission from the reaction zone was detected by a set of four PMTs (PMT1001, Thorlabs, Newton, NJ, USA) arranged together for 4-color pyrometry and detailed in previous work [13,15]. The PMTs were equipped with four bandpass filters with 10 nm pass widths and are indicated in Fig. 1. Light emission was collected using a reflective collimator (RC12SMA-P01, Thorlabs, Newton, NJ, USA) and passed through a 200 μ m optical fiber connected to the PMT-Pyrometer where radiation was recorded for further analysis. Using a single optical fiber to collect radiation ensured that the PMT signals originated from the same light emitters at any given time.

The choice of PMT wavelengths for pyrometry is generally compatible with light emission peculiarities during Al combustion [16]. When the thermite burns, the light emission of the Cu₂ molecule (at 500 nm) can be significant [17]. Then, unlike other PMTs that detected radiation emitted by the condensed phase, the PMT D signal was contaminated by the copper-originated contribution and was therefore excluded from data processing and further analysis. The contamination of the PMT D signal and its impact (if the signal is not excluded from data processing) is discussed in more detail in Supplementary Information.



Fig. 1. Sketch of the powder burning set-up. Circle inset provides a picture of the inside chamber with the thermite powder on the stand and the laser beam path shown.

4. Results and discussion

The PMT signals that were recorded in the experiment are presented in Fig. 2.

Following the formalism developed in Ref. 13, the temporal changes in the reciprocal temperature were calculated for the different combinations of PMT signals and are shown in Fig. 3 together with the average value. The plots were offset in the way that the reciprocal temperature change $\Delta T^{-1} = 0$ at 0.035 s for all combinations of PMTs. As explained in Ref. [13], this time is arbitrary, and its specific choice is determined by the user for clarity on the representation of the data.

Comparing the temporal behavior of the reciprocal temperature change and its standard deviation, which is presented in Fig. 4, we can see that the strongest spike in the reciprocal temperature change occurs at the same time as the strongest spike in the standard deviation. As stated in Ref. 13, such peculiarities of the reciprocal temperature indicate significant non-isothermality of the light sources contributing to the recorded signals at about 0.2 s, which requires further attention. Note



Fig. 2. Four discrete PMT signals labeled as A-D (with corresponding wavelengths shown in Fig. 1) from a representative thermite powder combustion test.



Fig. 3. Time-dependent reciprocal temperature change for different PMT combinations and its average value. The different line widths are shown to visually demonstrate that the graphs overlap.

that the standard deviation reported in Fig. 4 was calculated for the three combinations of the reciprocal temperature change shown in Fig. 3.

Fig. 5 shows the behavior of the PMT signals and the reciprocal temperature change around the strongest spike in the standard deviation.

The video of the process is provided in *Supporting Information* and shows that during the combustion experiment, the radiation was mainly emitted by thermite particles moving above the stand. Sometimes diffuse clouds appear for a short time, which further contribute to the glow.

Fig. 6 demonstrates an occurrence of such a cloud during the strongest non-isothermality, i.e., at the spike of the standard deviation of the reciprocal temperature change. The image difference in Fig. 6c was obtained by Python software [18] to isolate the cloud outlined in red (see Fig. 6b). Note that displacements of the thermite particles during the time between two processed frames lead to the appearance of twin



Fig. 4. Time-dependent standard deviation of the reciprocal temperature change calculated for three combinations of PMTs reported in Fig. 3.

circles in the difference image that are artificial.

The above analysis of the combustion progress allows us to conclude that the increase in the PMT signals in the time period between 0.205 s and 0.215 s reported in Fig. 5a is associated with the occurrence of the diffuse cloud. Then, by subtracting the local PMT background signal (i. e., the signal at 0.205 s) from the PMT signal during the time of interest,

we can obtain PMT signals that correspond to the bare light emission of the diffuse cloud. These subtracted PMT signals presented in Fig. 7 can be further processed to infer the reciprocal temperature change associated with the radiation emitted by the diffuse cloud.

This reciprocal temperature change exhibits different behavior depending on the time period, as shown in Fig. 8 and characteristic kinks are clearly distinguishable between the time periods. Unlike Figs. 5b and 7, Fig. 8 directly shows a distinct signature.

The plots corresponding to different PMT combinations are offset by different constants in different periods (i.e., I, II and III). In accordance with Eq. (4), such a change in displacement is an indicator of the occurrence of different physical states of the emitting material. Moreover, the almost constant temperature behavior immediately after transitions between periods, which can be interpreted as a consequence of the energy release during the corresponding transitions between the states, further confirms the above conclusion about different physical states. It is also worth noting that the order of the plots in Fig. 8, where "AB" is the highest and "BC" is the lowest, is the opposite of when the deviations in the plots are caused by the multi-temperature light emission (see Fig. 5b, for example). This provides further support for relating the observable difference to displacement due to phase changes.

The constant K in Eq. (1) is proportional to the effective area of the light emitters, the radiation of which is recorded by the PMT. Then, the ratio of the PMT signal to the Wien's function characterizes this effective emitting area. If we are only interested in the relative behavior of the effective emitting area, we can use the reciprocal temperature in the Wien's function instead of its absolute value. This type of analysis is like



Fig. 5. PMT signals (a) and reciprocal temperature change (b) at the time period of the strongest standard deviation spike shown in Fig. 4.



Fig. 6. Video frames captured at 0.205 s (a) and 0.207 s (b) together with their difference (c). The red circle shows a diffuse cloud whose occurrence is temporally correlated with the standard deviation spike shown in Fig. 4.



Fig. 7. PMT signals corresponding to light emission associated with the occurrence of the diffuse cloud shown in Fig. 6.



Fig. 8. Time-dependent reciprocal temperature change obtained by processing PMT signals (shown in Fig. 7) associated with the diffuse cloud radiation for different PMT combinations. Roman numerals indicate periods of time with noticeably different temperature behavior, which are separated by clearly visible kinks. Horizontal dashed lines at transitions between periods serve as a guide for the eye. They emphasize the almost constant temperature behavior immediately after the transitions between the time periods.

the relationship between reciprocal temperature and reaction rate derived from the Arrhenius law (see Eqs. (11), (12) in Ref. 13).

Fig. 9 shows the effective emitting area as a function of time, which was calculated using values corresponding to the light emission associated with the diffuse cloud, namely, the PMT B signal (brown line in Fig. 7) and the reciprocal temperature $\Delta T_{A,B}^{-1}$ (red line in Fig. 8). Note that the emitting area was normalized to the local maximum value at around 0.209 s, so it is reported in arbitrary units.

The behavior of the effective emitting area shown in Fig. 9, namely its monotonic increase with time, indicates that some condensed material is being generated within the time period of interest. In the video of the process, this condensed material is visible as the diffuse cloud that develops over time. Considering the chemistry of the system and the temperature level typically achieved during thermite combustion [17], it can be assumed that we observed the condensation of nanoalumina from gaseous aluminum suboxides. In the case of radiation emitted by corresponding nano-oxides, the effective emitting area is proportional to the volume of nanoparticles [19], and Fig. 9 could provide information about the evolution of the size of the growing nano-oxides. This analysis



Fig. 9. Effective emitting area calculated using values corresponding to the light emission associated with the diffuse cloud, namely, the PMT B signal (brown line in Fig. 7) and the reciprocal temperature $\Delta T_{A,B}^{-1}$ (red line in Fig. 8). It is normalized to the local maximum value at around 0.209 s and is reported in arbitrary units.

is beyond the scope of the current work.

Based on the assumption that radiation is emitted by nano alumina formed as a result of gaseous suboxide condensation, it becomes possible to identify the physical states shown in Fig. 8. The ascription of two states is quite obvious: these are the liquid (II in Fig. 8) and solid (III in Fig. 8) states of aluminum oxide. However, the state I in Fig. 8 is unique to condensing nanoparticles. We argue that this state corresponds to the supercritical clusters, the condensation growth of which is suppressed. Clusters become positively charged due to thermionic emission of electrons that effectively occur at high temperatures. These clusters, which were directly observed during magnesium combustion [20,21], appear due to a high energy release at the condensation stage, and therefore, should occur during nano alumina condensation as well.

The cluster cooling results in their loss of the positive charge further allowing them to eventually coalesce. Excessive surface energy released during coalescence [22,23], can preserve the temperature constant at the I-II transition (see Fig. 8). As noted above, such constancy of temperature is characteristic of a conventional phase transition. It is also seen in Fig. 8 for alumina solidification, i.e., the transition II-III. In addition, charged clusters (the state I) should have different electrical properties compared to uncharged nano alumina (the state II). In accordance with Eq. (4), this explains the change in the offset constants of the reciprocal temperature plots at the corresponding I-II transition (see Fig. 8).

The ascription of the II-III transition to the solidification of alumina, the temperature of which, i.e., 2345 K, is well-known [24], enables the inference of the absolute temperatures. Eq. (9) from Ref. [13] can be utilized with $T_0 = 2345$ K and applied to the measured reciprocal temperature changes. The absolute temperatures were then obtained as shown in Fig. 10.

In Fig. 10, the average temperature correlates well with the value reported previously for combustion of Al/CuO thermite [17]. It can be used to characterize the burning thermite particles. However, when the condensation of nano alumina occurs, this average temperature becomes misleading. As can be clearly seen, the maximum of the average temperature and the maximum of the actual temperature of condensing nanoparticles are noticeably different and are observed at different times, so the average temperature is not capable of describing the peculiarities of the process. The misleading predictions of traditional pyrometry have already been addressed in our original work [13] that introduced calibration-free pyrometry capable of characterizing multi-temperature light-emitting systems.



Fig. 10. Absolute temperatures obtained from the measured reciprocal temperature changes assuming that the II-III transition represents the solidification of alumina. "Subtracted" refers to the values corresponding to the subtracted PMT signals (see Fig. 7), i.e., to the light emission associated with the diffuse cloud. "Original" refers to the value processed using the recorded PMT signals (see Fig. 2). The inset shows the behavior of the average temperature over the entire combustion process.

Unfortunately, we are not able to report the exact value of the maximum temperature reached in the system, i.e., that at around 0.2055s. This requires knowledge of the light emission characteristics of charged clusters that occur in state I. The exact maximum temperature of clusters in state I could potentially provide further insight into the long-standing problem with the boiling point of alumina, whose value reported in the literature ranges from about 3250 K [25] to about 4200 K [26]. At the same time, it should be emphasized that there is a noticeably different temporal behavior of the temperature of the charged clusters (state I in Fig. 8) and uncharged liquid alumina nanoparticles (state II in Fig. 8). The cooling of charged clusters is described by a convex function, while cooling of uncharged growing nano-oxides is described by a concave function. An explanation of this difference will require a detailed consideration of various processes associated with the condensation growth of nano-oxides [10,21].

It is worth noting that when we talk about the temperature evolution of growing nano-oxides, we imply that the growth occurs simultaneously. This assumption is quite natural and is based on the sequence and hierarchy of the involved processes: nucleation (fast process) and condensation (slow process). In other words, nucleation occurs first at the highest supersaturation of gaseous suboxides, which leads to a drop in this supersaturation. After this, i.e., with less supersaturation and, therefore, suppression of nucleation, only surface growth is possible. Also, the rather sharp phase transitions observed in Figs. 8 and 10 supports all particles within the cloud undergoing the condensation process almost simultaneously and have similar temperatures at any given time. Otherwise, these phase transitions would be more smeared.

5. Concluding remarks

Condensation of gaseous metal suboxides is the main channel for the release of energy during the combustion of metals. Therefore, its time scale governs the overall energetic performance of reactive systems. Calibration-free pyrometry utilized to isolate the light emission that originates from nano alumina generated during the combustion of Al/CuO thermite, made it possible to reveal signatures of condensation processes in the reacting system. Direct observation of the temperature behavior of nano alumina over time and the identified transitions between different physical states of condensation mechanisms, which can inform accurate modeling of metal combustion processes.

CRediT authorship contribution statement

Nicholas R. Jaramillo: Writing – review & editing, Methodology, Investigation, Formal analysis, Data curation. Cole A. Ritchie: Software, Methodology, Investigation, Data curation. Michelle L. Pantoya: Writing – review & editing, Supervision, Project administration, Funding acquisition. Igor Altman: Writing – original draft, Supervision, Project administration, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Supplementary materials

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