



# Critical heat dissipation length scales in fully dense thermite foils

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## ABSTRACT

One strategy to suppress metal vapor production in fully dense thermite mixtures is to dilute with excess inert metal to lower the reaction temperature below the boiling point of the metal. However, if the diluent is inhomogeneously distributed throughout the microstructure, localized hot regions can be present, resulting in local temperatures exceeding the boiling point of the metal. Fully dense Al:Cu<sub>2</sub>O:Cu foils are analyzed to determine the critical length scales necessary to suppress Cu vapor during reaction. The microstructures of these foils, more specifically length scales of reactive and inert material, are critically analyzed using image analysis. The results from image analysis are then compared with finite element heat transfer simulations used to determine critical length scales of thermite and diluent required to suppress metal vapor production during reaction of fully dense Al:Cu<sub>2</sub>O:Cu foils.

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## 1. Introduction

Thermite was originally developed over 100 years ago to produce molten metal capable of brazing metal components [1,2]. In a thermite reaction, a metal oxide is reduced by a metallic fuel, typically aluminum, releasing substantial amounts of heat, often enough to melt and partially vaporize the resulting metal. The ability to produce heat and vapor [3–8] has led researchers to explore propulsion applications of nanoenergetic thermite reactions [9,10], including the less reactive Al:Cu<sub>2</sub>O system studied in this paper [11]. Despite the long history of thermite development and the wide variety of applications, the prediction and control of gas release during self-propagating thermite reactions is still a challenging endeavor.

Gas generation often occurs due to the vaporization of metal during propagation, as well as the release of oxygen during oxide decomposition [11–13]. Diluting thermite mixtures with inert material can suppress gas formation [14,15] because the inert material absorbs the heat released by the reaction, thereby decreasing the reaction temperature and the chance of vaporization. Gas suppression via dilution has been demonstrated in our previous work with mechanically fabricated Redox Foils [16,17], as well as with multilayers made with physical vapor deposition (PVD), where the spacing of species can be controlled systematically [18].

Most thermite materials are produced via powder processing methods, and therefore are not in a fully dense form, even when

pressed into green compacts [19,20]. The inherent porosity in the compacts provides channels for evolved or trapped gases to expand. The production of vapor, as well as the rapid heating of confined gases, can create a pressure wave capable of expelling reacting thermite material away from the reaction front, further enhancing the transfer of mass and heat. This advection of molten material has been found to be a contributing factor in large flame velocities, especially in confined experiments [21,22].

Despite the complex and dynamic nature of self-propagating thermite reactions (and formation reactions), researchers have studied thermal transport during the ignition and combustion of reactive materials. For example, Ward et al. [23] numerically modeled the heat transfer that leads to ignition when metal powders are heated rapidly on a wire. Their transient heat transfer model is based on a finite difference method and accounts for the temperature gradient across the thickness of the powder compact on the wire and the conductive heat losses to the powder. In a separate attempt, Bajiot et al. [24] developed a theoretical model to predict gas generation in thermite reactions based on the extent of reaction, assuming the reaction progresses in thermodynamic equilibrium. When applied to a variety of commonly used aluminum-based thermite systems, though, the resulting predictions showed poor agreement with experimental observations [24]. An updated micro-kinetic model incorporating condensed and gas phase reactions of the Al:CuO system, yielded predictions that match experiments more effectively [25]. Another study by Knapp et al. [26] used a hot-spot-model to study ignition and combustion of the Al:CuO system. The model assumed temperature-independent specific heat capacity and heat conductivity to simplify the calculations, and confined the rate limiting step to the decomposition of

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the oxide. The authors acknowledged that these assumptions and simplifications may explain discrepancies between calculated and experimental propagation rates. Experimentally determined velocities were compared with the calculated reaction rates, identifying significant discrepancies between the measured and predicted velocities for fuel-rich conditions. Together, these various numerical modeling efforts focused on gas producing systems with low densities. None of them examined condensed-phase heat transfer during the self-propagation of thermite reactions in fully dense materials. Here we do so to study the effect of microstructure length scales on temperature uniformity and the desired suppression of metal vapors as heat is dissipated from reacting thermite regions into regions of an inert diluent.

Our recent work is focused on developing a fully dense thermite foil for bonding applications [16,17] using the Al:Cu<sub>2</sub>O thermite system diluted with inert Cu. One key impediment to the implementation of such a thermite system for bonding is the production of gaseous vapor during reaction propagation [16,27]. The Al:Cu<sub>2</sub>O system is favored over the Al:CuO system [3] as it yields less gas while also producing more molten Cu product, which can be used as a braze. However, the Al:Cu<sub>2</sub>O must be diluted with greater than 18 weight percent Cu for the adiabatic reaction temperature, a thermodynamic calculation that assumes equilibrium reactions, to drop below the boiling point of Cu [16].

The thermite or Redox Foils considered here can be fabricated using arrested reactively milled (ARM) thermite powders, combined with diluent powders. During the ARM processing of nanocomposite thermite powders, the reactant powders are ball-milled to reduce the spacing between the fuel and the oxide by continually fracturing and cold welding particles together. This reduction in reactant spacing increases the rate of heat production by decreasing the mass diffusion distances required to complete the reduction–oxidation reaction. However, milling is arrested before powders react exothermically in the milling vial [28–31]. Once the composite thermite powders are prepared, they are compacted in steel tubes and then swaged to full density. Finally, the tubes are cold-rolled to produce flat strips and the steel jackets are removed to yield dense thermite (Redox) foils [16,17,32].

In our previous Redox Foil work, two variations in the fabrication method were used to produce Al:Cu<sub>2</sub>O:Cu foils, resulting in different local distributions of thermite and diluent regions. In the first strategy, nanocomposite thermite powders (Al:Cu<sub>2</sub>O) were fabricated via ARM and Cu diluent powders were added before mechanical consolidation and refinement. In the second strategy, all three constituent powders (Al, Cu<sub>2</sub>O, and Cu) were milled together before consolidation and refinement [17]. The first approach produced Cu vapor during propagation, despite thermodynamic calculations indicating that reaction temperatures should be below the boiling point of Cu. The lack of Cu vapor suppression is attributed to a coarse spacing of the Al:Cu<sub>2</sub>O and Cu regions. The coarse spacing limits the Cu's ability to absorb heat rapidly enough to avoid high temperatures and boiling in the local Al:Cu<sub>2</sub>O thermite regions. The second approach, of milling all constituents together, provided a more uniform distribution of thermite and diluent regions and thereby suppressed Cu gas production.

The goal of this study is to understand what combination of dilution limits and thermal diffusion length scales are required to suppress Cu vapor formation during the reaction of Al:Cu<sub>2</sub>O:Cu Redox Foils. Although the thermodynamic dilution limit for vapor production can be easily calculated, the critical length scales to suppress localized hot spots during the reaction are unknown. The microstructure of foils fabricated with ARM thermite powders and Cu diluent powders is first characterized to determine the distribution of thermite and diluent spacings. This microstructural characterization is then compared with finite element heat transfer simulations that are used to predict the critical thermite and diluent

lengths that are necessary to suppress Cu vapor production during self-propagation of reduction–oxidation reactions in fully dense foils.

## 2. Microstructural characterization

The fully dense Redox Foils analyzed in this paper were produced by cold swaging and rolling a mixture of nanocomposite Al:Cu<sub>2</sub>O thermite powders and Cu diluent powders. The foil fabrication process is described elsewhere [16,17,32], and the foils analyzed in this work were experimentally characterized in our previous paper [17]. Briefly, the ratio of Al and Cu<sub>2</sub>O powders is maintained at the 2:3 stoichiometric ratio, and the quantity of diluent is defined as a weight percent of the total foil mass. Here we only address the critical length scales for Al:Cu<sub>2</sub>O:Cu ARM foils produced with Cu dilutions of 20 wt%, 30 wt%, and 40 wt%. Based on previous experimental characterization, the 20 wt% and 30 wt% produce gaseous Cu during propagation, whereas the 40 wt% foil does not as determined by emission spectroscopy on the light emitted from the self-propagating reaction.

As-fabricated foils were mounted in epoxy and imaged with a Tescan Mira GMU III SEM using a backscatter electron detector to enhance compositional contrast. To confirm elemental species, energy dispersive X-ray spectroscopy (EDS) mapping was performed using an EDAX Element detector as presented in Fig. 1. Large areas of the microstructure were characterized using the Tescan stitching function that combines 64 individual images to create a “stitched” image with a 250 μm field of view. The stitched image retains the scale of the individual images which were captured at 8000× magnification at a scale of 33.789 nm/pixel. An example of a stitched image is provided in Fig. 2. More specific details of the image acquisition process are provided in the supplementary information. Four stitched images were acquired and analyzed for each dilution. Each stitched image was captured on a different part of the foil to make sure local variations were not oversampled for each level of dilution.

To assess the heat dissipation length scales, the images were analyzed using code developed with the Matlab Image Analysis Toolbox. A detailed explanation of the algorithms is provided in the supplementary information. In summary, the algorithms threshold the image into thermite regions and diluent regions, then subsequent processing finds the local ratio of thermite length to diluent length, to be referred to as the ratio. Calculation of the ratio of thermite to diluent assumes a planar geometry, just as the thermal transport model explained in the next section. This is a reasonable assumption considering that the extensive rolling causes the diluent to flatten into a plate-like morphology. The ratio describes the local level of dilution, with higher ratios corresponding to smaller local dilution. Correlations between the ratio and equivalent weight percent dilution (from the fabrication process) are provided in Fig. 3.

## 3. Thermal transport model

We use a thermal transport model to predict the thermal response of experimentally developed Redox Foils by systematically changing the length scales of thermite and diluent. Five different thermite length scales were simulated: 1 μm, 5 μm, 10 μm, 15 μm, and 25 μm, where the thickness of the diluent length scales is defined by the level of dilution. Four levels of dilution were simulated for each length scale: 10 wt%, 20 wt%, 30 wt%, and 40 wt%. A summary of the simulated lengths is provided in Table 1.

The generalized thermal diffusion equation with a heat source is described by:

$$\rho C_p \frac{\partial T}{\partial t} + \nabla \cdot (-\kappa \nabla T) - \dot{Q}_{src} = 0, \quad (1)$$

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