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# The utilization of metal/metal oxide core-shell powders to enhance the reactivity of diluted thermite mixtures

Karsten Woll<sup>1</sup>, John David Gibbins, Kyle Slusarski, Alex H. Kinsey, Timothy P. Weihs\*

Department of Materials Science and Engineering, Johns Hopkins University, Baltimore, MD 21218, USA

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## ABSTRACT

When thermite mixtures combust considerable amounts of gas can be released which impedes their application in the field of joining. Diluting the thermite mixture with excess metal can lower the reaction temperature and thereby minimize mass ejection. However, increasing the degree of dilution reduces reaction efficiency. This paper demonstrates that the reduction of reaction efficiency is significantly less for the Al/NiO thermite system if the excess metal (Ni) is added using a fuel/core-shell mixture in which Al fuel particles are mixed with Ni/NiO core-shell particles. The use of Ni/NiO core-shell particles reduces the average distance for aluminum and oxygen intermixing by placing the fuel (Al) in direct contact with the oxide (NiO), separate from the diluent (Ni). Mixtures comprised of Al and Ni/NiO core-shell powders were fabricated, characterized, and compared to compacts fabricated using conventional Al, NiO, and Ni powders for dilutions up to 40 wt% Ni. The fuel/core-shell particle geometry increased combustion front velocities for all Ni dilutions. Mass ejection decreased uniformly for both particle geometries as the degree of dilution increased.

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## 1. Introduction

The reduction-oxidation (redox) reaction between a metal oxide and Al that forms a molten metal and alumina has been utilized in joining applications for nearly 100 years [1,2]. The classic application is the welding of railroad lines [3,4]. In this particular case iron oxide reacts with Al and the molten iron product flows out of a crucible and into a mold surrounding the joint. The less dense alumina floats to the top of the crucible and is discarded. More recent studies have demonstrated that copper-based thermite reactions can enable welding of copper conductors under reduced gravity in space [5], and self-propagating thermite reactions have been observed in thin multi-layered foils [6–11]. The combination of a foil geometry and the release of large heats and molten products suggests there is potential for utilization of thermite reactions as very local heat sources for joining. When placed directly between two components, such foils may transfer the traditional railroad line welding application to a smaller scale as well as enable other types of joining such as soldering and brazing [12]. Unlike welding in which one melts the components being bonded, in soldering and brazing, a filler material melts and wets the

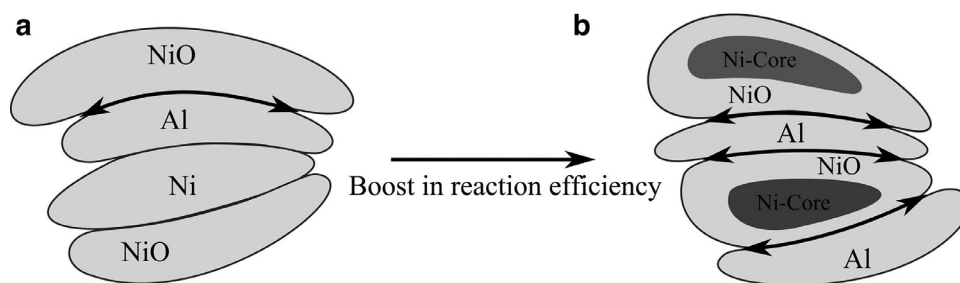
components to enable bonding [13]. The components, themselves, do not melt. In the case of soldering, the filler material turns liquid below 450 °C and for brazing the filler material melts above 450 °C.

Soldering with reactive foils that are based on formation reactions is an established commercial process called NanoBond™ [14,15]. These foils release heat based on the formation of intermetallic compounds, and melt pre-existing solder layers on to that have been placed on the components. The redox reaction inherent to thermite-base reactive foils offers two significant advantages for joining compared to intermetallic formation reactions. First, the higher reaction temperatures of the redox reactions make them more capable of enabling brazing of components. Second, the molten metal produced by the redox reaction can act as a filler (braze) material, thereby eliminating the need for pre-existing solder or braze on the components being bonded. It is anticipated that these advantages of the redox reactions have the potential to broaden the range of joining applications that can be addressed with reactive foils acting as local heat sources.

While redox reactions produce substantial heat and a molten braze, most of these reactions also produce large amounts of gas [16,17], leading to material ejection and detrimental pore formation during bonding. Both of these factors limit joint strength. Consequently, the utilization of redox reactions in joining applications requires a significant reduction in gas release. This study explores minimization of gas production in the Al/NiO thermite system, a

\* Corresponding author. Fax: +1 4105165293.

E-mail address: [weihs@jhu.edu](mailto:weihs@jhu.edu) (T.P. Weihs).<sup>1</sup> Present address: Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), 76344 Eggenstein-Leopoldshafen, Germany.



**Fig. 1.** Schematics showing the use of (a) elemental Ni powders or (b) core-shell, Ni/NiO powders in the formation of Al/NiO–Ni thermite foils with excess Ni. The Al/NiO reactive interfaces are indicated by black arrows.

chemistry for which gas release is already limited [17]. The main factors thought to determine the degree of gas generation and the peak pressure for a given system are the heat of reaction,  $\Delta H_{rx}$ , and the combustion front velocity,  $v$  [18].  $\Delta H_{rx} = 3445$  J/g [17] for the Al/NiO system and leads to an adiabatic reaction temperature,  $T_{ad}$ , of 2914 °C [17] and gaseous Ni in the reaction products. Researchers have shown that they can affect  $\Delta H_{rx}$ ,  $T_{ad}$ , and  $v$  by varying the geometry or the composition of the thermite mixture and, in doing so, can reduce gas generation [18–25]. Researchers reported that three parameters can substantially reduce the amount of gas released: 1) increasing the particle size [18,21,22] 2) altering the fuel/oxide stoichiometry [20,23], and 3) adding inert diluents, such as oxides and metallic elements [24,25]. Minimization of gas production with excess metal is the focus of the current study because the excess metal has the added benefit of increasing the percentage of braze in the final bond area.

Recent studies have reported on the addition of excess metal in loose thermite mixtures with heterogeneous microstructures [26–28]. Shen et al. studied the addition of nanosized Ag powder to the Al/CuO system [26]. They diluted this thermite mixture up to 10 wt% Ag and observed that front velocity initially increased, peaked at 2 wt% dilution, and then decreased substantially on approaching 10 wt% Ag. The initial rise in combustion front velocity is attributed to silver's ability to increase the mixture's thermal conductivity. At higher dilutions, though, the drop in the total heat of reaction dominates over the improved heat transfer and reaction front velocity drops significantly. In a related study, Bohlouli-Zanjani et al. investigated the effects of Cu dilution on two redox systems: Al nanopowder and NiO nanowires and conventional Al/CuO powder mixtures [28]. The dilutions range from 50 wt% to 80 wt% Cu, and the mixtures were all very fuel-rich. They examined phase transformations up to 800 °C under slow heating conditions and the associated heat releases. However, the propagation behavior was not studied. Alumina, AlNi, and Cu were the main reaction products after heating the Al-rich Al/NiO mixtures to 800 °C. The measured  $\Delta H_{rx}$  dropped by a factor of 5 when the amount of excess Cu was increased from 50 to 80 wt%. They found an even stronger effect of excess Cu in the Al:CuO thermite system. Bohlouli-Zanjani et al. noted that the excess Cu decreased reaction efficiency by creating relatively nonreactive Al/excess metal interfaces [28]. At the same time, the addition of excess Cu reduces the density of highly reactive Al/NiO interfaces. Such geometric effects are expected whenever diluent powders are added to compacts of fuel and oxide powders and they can lead to lower reaction front velocities and the risk of reaction quenching, particularly for heavily diluted systems.

This paper explores a core-shell powder geometry in the Al/NiO thermite system that demonstrates a significant boost in reaction efficiency and hence a lower chance of quenching as excess Ni is added as a diluent. The basic concept is demonstrated schematically in Fig. 1. When diluting a thermite mixture the excess metal can act as a barrier to oxygen transfer. If Ni is added as an

elemental powder to the Al/NiO mixture, Al/Ni interfaces form in addition to the desired Al/NiO interfaces that are most reactive (see Fig. 1(a)). Hence, the density of the reactive Al/NiO interfaces is reduced by dilution and reaction efficiency drops; further still, the average spacing between the fuel (Al) and the oxide (NiO) increases. In order to prevent the formation of Al/Ni interfaces and to minimize the decrease in reaction efficiency when diluting, compacts using Ni/NiO core-shell particles were fabricated. These particles comprise NiO as the outer shell and elemental Ni diluent as the inner core (see Fig. 1(b)). In this case, it is expected that Al particles exclusively form interfaces with the oxide and not the diluent (Ni). Consequently, the density of the reactive Al/NiO interfaces is substantially increased causing a boost in reaction efficiency. As a side effect, a substantial reduction in the average spacing of reactants is anticipated, which in turn will speed the rate of atomic mixing and combustion front velocities. Both the boost in reaction efficiency and the reduction in average reactant spacing should decrease the risk of quenching for a redox reaction that is heavily diluted to minimize gas production and therefore should open the possibility of bonding with thermite reactions.

## 2. Experimental

### 2.1. Sample preparation

An ideal geometry for utilizing a redox reaction for joining is a fully dense foil, measuring 100–500  $\mu\text{m}$  thick, with a fine continuous or discontinuous layering of reactants. There are two common approaches for fabricating thermite foils with fine reactant spacings: bottom-up deposition techniques [29–31] and top-down mechanical processes [32–36]. Deposition techniques are known to produce reactive foils with well-defined layering and total thicknesses. Mechanical processes offer less control of reactant spacing and foil thickness but do have the potential to produce thermite foils at lower costs and in higher volumes. Hence, the reactive foils for this study were fabricated using low cost powders and a combination of inexpensive mechanical processes that include compaction, swaging and rolling.

Rotary swaging is a mechanical processing method in which a rod or tube is rapidly compressed through a series of radial reductions. The swager itself contains a large flywheel that rotates a roll cage with roller bearings over different interchangeable dies with varying diameters. In contrast to cold-rolling, there is close to 100% materials utilization, and the amount of microstructural refinement can be controlled by the total reduction of the rod's diameter. A successful utilization of swaging to fabricate Ni/Al reactive powders was recently demonstrated in [36].

After mixing the powders thoroughly, they were poured into carbon steel tubes in small increments to avoid segregation and compressed under normal stresses equal to 40 MPa. The compacted tubes were then swaged from an outer diameter of 15 mm down to an outer diameter of 3.2 mm (referred to as powder-in-tube

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