# Properties of reactive Al:Ni compacts fabricated by radial forging of elemental and alloy powders

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## ARTICLE INFO

## ABSTRACT

We explore rotary swaging of powders into solid compacts as an inexpensive means of producing reactive materials with refined microstructures and improved properties. Rotary swaging is a cold forging process that reduces the diameter of tubes, and in this study the tubes are packed with reactive combinations of elemental and alloy powders. The diameter reductions create a nearly fully dense compact from the powders and also reduce the average reactant spacing through plastic deformation. The extent of diameter reduction controls the microstructural refinement, observed through cross-sectional imaging. We correlate the observed changes in microstructure with reaction properties that are characterized using differential scanning calorimetry (DSC), hot-plate ignition studies, and velocity measurements. Exothermic peaks in DSC scans all shift to lower onset temperatures; hot plate ignition temperatures decrease; and reaction velocities rise as the degree of swaging is increased. We vary the shape of the initial reactants by substituting Ni flakes for Ni powders and find no improvement in microstructure or reaction properties, due to clumping of the Ni flakes during the initial compaction steps. We also vary chemistry by substituting Al-Mg alloy powders for Al powders and find that the alloy powders yield lower DSC exothermic peak temperatures, lower ignition temperatures, and higher reaction velocities compared to similar compacts with pure Al powders. This combination of results suggests that rotary swaging is an effective technique for producing reactive materials at low cost.

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## 1. Introduction

Exothermic formation reactions that generate intermetallic compounds and heat have been studied extensively [1–8] and can occur in compacts of homogeneous [1–8], composite [9–28] or core shell particles [29–31], or in layered films [32–40] or foils [41–45]. These reactive materials (RMs) can be fabricated either through a bottom-up deposition method or using a top-down mechanical process. Most deposition methods utilize physical vapor deposition (PVD), such as magnetron sputtering, and allow for precise control of the average reactant spacing on a nanometer scale [32–40]. However, such processes are more expensive than mechanical methods due to high capital costs and lower throughput on a per mass basis. Mechanical methods for fabricating RMs include ball-milling [9,11–19,21–28], rolling [10,41–45], and cold isostatic pressing [3]. These methods typically have lower capital costs and higher throughputs but cannot achieve the same microstructural uniformity as PVD methods. However, ball milling is capable of fabricating composite powders with reactant spacing on the scale of 10's of nanometers [9,11–19,21–28].

Rotary swaging is an alternative mechanical processing method with low capital costs, inexpensive starting materials, and high throughput. The swaging process works by rapidly compressing a rod or tube through a series of radial reductions [46–49]. The swager itself contains a large flywheel that rotates a roll cage with roller bearings over different interchangeable dies with varying diameters as shown in Fig. 1(a). As the roller bearings pass over the dies, the dies rapidly compress against a tube or rod, causing it to condense to the desired outer diameter (OD) as shown in Fig. 1(b). Each set of dies introduces a given average strain to the sample, and the number of dies and the strain for each set of dies can be varied. The radial nature of the deformation minimizes fracture within the sample and thus leads to higher materials utilization compared to cold rolling. The large plastic strains imparted by the dies act to shear the constituent powders, breaking surface oxides and enhancing bonding between powders. This is important for compacts containing Al powders.

With rotary swaging, one can control the degree of microstructural refinement and hence the reactivity of the resulting powder compacts in multiple ways. One is through the total reduction of the
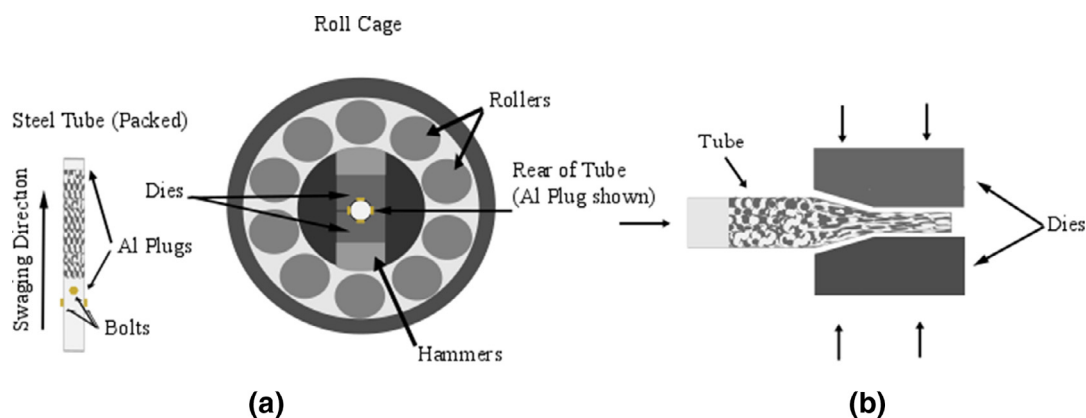
**Fig. 1.** Schematic diagrams of a 3Fenn Rotary Swager. (a) A front view of the swager showing two dies coming together to deform a metal tube loaded with powder. During operation the swager spins a roll cage lined with rollers that pass over a set of hammers that compression the dies together. (b) A cross-sectional view of a tube being deformed to a smaller diameter by two dies as it is pushed into the swager.

**Table 1**
Chemistries, particle geometries and average reactant spacings for the five compacted samples. (P = particle, F = flake, and LP = laminate particle.) The average reactant spacing is determined using the lineal intercept method where the Al/Ni or Al-Mg/Ni boundaries determine intersections, and the average spacing is equivalent to ½ of the average reactant width.

|  | Compact chemistry (at%) | Initial powder shape and size (mesh) | Average reactant spacing (μm) for 6.35 mm samples | Average reactant spacing (μm) for 2.54 mm samples |
|---|---|---|---|---|
| Al:Ni P/P | 50 Al<br>50 Ni | Al: sphere: -325<br>Ni: sphere: -325 | 3.16 ± .42 | 2.01 ± 0.31 |
| Al:Ni P/F | 50 Al<br>50 Ni | Al: sphere: -325<br>Ni: flake: -325 | 6.17 ± 2.06 | 1.82 ± 0.20 |
| Al-Mg:Ni P/P | 46 Al<br>4 Mg<br>50 Ni | Al-Mg: sphere: -325<br>Ni: sphere: -325 | 4.11 ± 1.06 | 1.57 ± 0.31 |
| Al-Mg:Ni P/F | 46 Al<br>4 Mg<br>50 Ni | Al-Mg: sphere: -325<br>Ni: flake: -325 | 8.38 ± 2.71 | 1.99 ± 0.28 |
| Al:Ni LP | 48 Al<br>52 Ni | Al:Ni: sphere: 212–355 μm<br>laminate structure | 0.35 ± 0.08 | 0.15 ± 0.02 |

rod's diameter with larger reductions leading to more refinement. A second is through the size of the starting powders with smaller initial powders leading to finer and more reactive as-fabricated microstructures, as seen in earlier powder compacts [7]. A third is through the use of composite particles such as ball-milled, laminate or core-shell powders. Lastly, alloying the elemental particles can also enhance the reactivity of the final compacts. To examine all four factors, we characterize the microstructures and properties of reactive compacts that were swaged from elemental powders (Al and Ni), alloy powders (Al-Mg), Ni flakes, and laminate powders (Ni:Al), and the effects of swaging reduction, initial particle geometry, composite powders, and reactant chemistry are assessed.

## 2. Experimental procedures

### 2.1. Fabricating materials

Five different samples were fabricated using various powder chemistries and geometries as shown in Table 1: (a) Al powders and Ni powders (Al:Ni P/P), (b) Al powders and Ni flakes (Al:Ni P/F) (c) Al-Mg powders and Ni powders (Al-Mg:Ni P/P) (d) Al-Mg powders and Ni flake (Al-Mg:Ni P/F) and (e) Ni:Al laminate powders (Al:Ni LP). The single phase powders and flakes were purchased commercially (Goodfellow and Alfa Aesar) and were mixed to achieve a 50:50 Al:Ni or Al-Mg:Ni chemistry, and the laminate powders were fabricated in-house through a combination of rolling and grinding to achieve a 52:48 Al:Ni chemistry and an average reactant spacing of 1.5 μm [10]. The 52:48 and the 50:50 Al:Ni mixtures are expected to form

the B2 NiAl intermetallic upon reaction, based on the Al:Ni equilibrium phase diagram. An Al-8 at%Mg alloy powder was also chosen as a replacement for the Al powder for two reasons. First, it is a dual phase alloy with a lower melting temperature than pure Al. (The eutectic melting temperature is only 450 °C on the Al-rich side of the Al-Mg phase diagram). The lower melting temperature should speed mixing of Ni into the Al-Mg reactant because it will melt earlier in the reaction process as the reaction self-propagates by a given location. Second, the two-phase alloy will be harder than pure Al due to precipitation hardening and should force more deformation in the Ni phase during the swaging process [50–51].

An Instron 5582 load frame was used to compact mixed, loose powders into 15 mm OD stainless steel tubes to 39.3 MPa. The process of mixing and packing was completed in five steps using 20 g quantities in each step so as to minimize segregation of powders. The calculated theoretical density after compacting was approximately 60%. The starting tubes, which were ~40 cm in length, were plugged at both ends with Al rods to keep the powder from spilling out during swaging. The bottom rod was ~20 cm long while the top plug was only 2.54 cm long. Each tube had its diameter reduced from 15 to 6.35 mm as shown in Fig. 1(a) through a series of 5 reductions and to 2.54 mm through a second series of 5 reductions, all with a Fenn 3F rotary swager. After reaching a 6.35 mm OD, approximately 45 cm were cut from the front end of the ~ 60 cm long tube. The remaining sample was swaged to a 2.54 mm OD. After swaging, the steel jackets around the samples were removed by milling one side of the rod and peeling off the jacket. Once free of the jackets, samples were cut into 3 sections to explore variations in density along the length of the