



# Facile synthesis of micro-sized Ni–Al alloy powders through low-temperature chemical alloying

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## ABSTRACT

Micro-sized Ni–Al alloy powders were synthesized using different quantities of Ni and Al powders as well as AlCl<sub>3</sub> as an activator in a quartz batch reactor at  $\leq 773$  K. This method, named low-temperature chemical alloying (LTCA), which allows Al atoms to diffuse into micro-sized nickel particles mediated by aluminum chloride (chemical promotor), is distinct from conventional processes, such as cast-and-crush, and gas atomization. As-synthesized Ni–Al alloys were characterized using different analytical techniques that included X-ray diffraction (XRD), particle size analysis, and field emission scanning electron microscopy in conjunction with energy dispersive X-ray spectroscopy (FESEM-EDS), to confirm the formation of alloy phases, such as Ni<sub>3</sub>Al, NiAl, Ni<sub>2</sub>Al<sub>3</sub>, and NiAl<sub>3</sub>. The analytical results showed that the crystalline phase compositions of the Ni–Al alloys were highly dependent upon the initial amounts of Ni and Al powders employed at the given alloying conditions (alloying temperature, 773 K; alloying time, 20 h; amount of AlCl<sub>3</sub>, 1.2 wt%). As a result of the thermal treatment of Ni–Al powder mixtures with the Al contents of (5, 15, 30, and 50) wt% under continuous powder mixing by rotation, each powder was found to have (i) Ni solid solution + Ni<sub>3</sub>Al, (ii) Ni<sub>3</sub>Al, (iii) NiAl, and (iv) Ni<sub>2</sub>Al<sub>3</sub> + NiAl<sub>3</sub> phases, respectively, corresponding to the equilibrium states of the Ni–Al phase diagram. The cross-sectional analyses showed that the alloy structures of the heat-treated powders exist in a single-phase or core-shell form, depending on the number of crystalline phase compositions predicted from the phase diagram. In particular, the Ni–50 wt% Al powder has a unique Ni<sub>2</sub>Al<sub>3</sub>@NiAl<sub>3</sub> core-shell structure. We further evaluate the performance of the as-developed Ni–Al alloy powders as oxidation-resistant materials and template materials for high-surface area ( $\sim 60$  m<sup>2</sup>/g<sub>cat</sub>) nickel catalysts.

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## 1. Introduction

Ni–Al binary alloys have been widely employed in numerous fields due to their superior properties, such as high temperature oxidation and creep resistance, high specific strength, low density,

and high catalytic activity [1–7]. There are five thermodynamically stable alloy phases (Ni<sub>x</sub>Al<sub>y</sub>) in the temperature range of 673–913 K in the Ni–Al binary system, namely (i) Ni<sub>3</sub>Al, (ii) Ni<sub>5</sub>Al<sub>3</sub>, (iii) NiAl, (iv) Ni<sub>2</sub>Al<sub>3</sub>, and (v) NiAl<sub>3</sub>, as shown in Fig. S1 of the Supplementary Information (SI) [8]. Applications of these Ni<sub>x</sub>Al<sub>y</sub> alloys are diverse, depending on their relative Ni/Al ratios. For example, Ni<sub>3</sub>Al or NiAl, which possesses ordered face-centered cubic L1<sub>2</sub> (Ni<sub>3</sub>Al) or base-centered cubic B2 (NiAl) crystal lattices, respectively, is employed for applications where high-temperature mechanical properties are required (e.g., aircraft turbine blade and aerospace industry), because these alloys exhibit higher melting points and excellent oxidation resistance [9]. In addition to the aforementioned Ni-rich

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