



# Study on the ignition mechanism of Ni-coated aluminum particles in air



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## ABSTRACT

Since aluminum responds to various oxidizers and has a high energy density, there are high expectations for its usefulness as a fuel. However, it is covered with an aluminum oxide film, which has a high melting point, and thus, its ignition is difficult. One method suggested to solve this problem is nickel coating; however, in contrast to the extensive amount of research conducted on the overall phenomenon of aluminum combustion, research regarding Ni-coated aluminum is still in nascent stages. This study was carried out to further elucidate the ignition mechanism; thus, millimeter-sized (~2.38 mm) aluminum particles were used to observe the surface where ignition occurs in air. The spatial and temporal resolutions were heightened by prolonging the heating period. The aluminum particles were nickel coated using electro/electroless methods, and surface analysis by SEM, thermal analysis by TGA/DSC, and species analysis by XRD and EDS were carried out. In addition, two-wavelength pyrometry was used to measure the ignition temperature. The results show that regardless of the nickel content in the coating of the aluminum particles, the ignition temperature was approximately 2400 K, similar to the melting point of aluminum oxide. The thermodynamic and thermophysical characteristics of nickel, aluminum, aluminum oxide, and nickel (II) oxide, and the surface/cross-sectional analysis, thermal and species analysis, and high-speed cinematography of the quenched samples provided a detailed explanation of the ignition process. Through this ignition mechanism, the emitted spectrum of AlO (as an intermediate combustion material) was traced to explain the decrease in ignition delay with increase in nickel content.

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## 1. Introduction

Aluminum ( $\text{Al}_2\text{O}_3$  as a metal oxide,  $\Delta H_f^\circ_{298} = -1,676 \text{ kJ/mol}$ ) has a high energy density and has been used as an additive in solid propellants for a long time [1,2]. In addition, it has been researched as a fuel for underwater propulsion systems that use seawater [3–6] and is important as a fuel for lunar exploration and Mars missions [7,8] and for power generation and hydrogen production from reaction with water [9]. Aluminum's high-temperature combustion is also considered important in the self-propagating high-temperature synthesis (SHS) of materials and in the production of metal oxides [10].

Aluminum (Al) is also used as a dust material that causes secondary explosions in thermobaric weapons. The primary explosion scatters Al in the air in a dust cloud where the secondary explosion occurs. This explosiveness depends on the combustibility,

ignitability, oxidizability, dispersivity, and confinement of the dust cloud. In thermobaric weapons, the explosives have an excess of Al fuel; thus, a large part of the Al propagates into air, is exposed to additional oxygen in the air, and after-burns for complete combustion [11,12]. The energy released through after-burning lengthens the duration of the blast overpressure and enlarges the fireball [12,13]. The explosive contains a considerable number of Al particles to increase the strength of the blast overpressure, and the amount of emitted energy depends on these Al particles [12–14]. Therefore, in thermobaric weapons, numerous Al particles inevitably meet air as an oxidizer, enabling successful ignition, which is vital for complete combustion. Consequently, it is necessary to improve the ignitability of Al in air to render it more combustible, for which a deep understanding of this phenomenon is required, thus a lot of research on ignition and combustion of cloud, agglomerates and suspensions of Al particles in flames [15–17].

However, Al has a disadvantage in that it naturally oxidizes forming an  $\text{Al}_2\text{O}_3$  film with a high melting point and is therefore difficult to ignite. Furthermore, the oxide film causes

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agglomeration of Al within solid rocket motors during combustion and forms a slag that deters steady burning [1]. Therefore, it is difficult to directly use Al as a fuel. To overcome such problems, Yagodnikov suggested three methods: volume modification of Al by chemically active elements, complete removal of  $\text{Al}_2\text{O}_3$  from the particle surface, and application of a controlled-property coating to the particle surface [18]. Various research groups have been interested in the third method, and Ni has been the most commonly used coating material for this purpose [18–31]. Other groups used copper [30], carbon [32], and iron [23,29] as a coating material. The reason for mainly using Ni as a coating material is its corrosion resistance. A thin coat of Ni on an Al surface prevents Al oxidization during general storage conditions and an initial warming phase [18]. In addition, the melting point of Ni (1728.15 K) is higher than that of Al (933.45 K); hence, Ni provides thermophysical protection at high temperatures. Nevertheless, good ignitability can be expected from Ni because its melting point is much lower than that of Al oxide (2325 K) [33]. Moreover, it has been reported that when an Al surface is coated with Ni, agglomeration of unburned Al, which hinders burning, is reduced [20–22,30,34].

Most of the previous studies have focused on  $\text{CO}_2$ , Ar, the burned gases of propellants, and clouds of Al particles or powder pack combustion in an air environment, according to their research purpose and experimental environment. Therefore, research on ignition mechanisms has been limited to single particles in  $\text{CO}_2$  and Ar environments [15,23,25,28,32]. Recently, Houim et al. carried out an experiment on the ignition of micron-sized Ni-coated and uncoated Al in the post flame of a multidiffusion flat-flame burner [35]. They showed that the ignition temperature of Ni-coated Al decreased in the post flame; this result is same as that obtained by other researchers. They further explained that the combustion characteristic was not affected by Ni coating despite the decrease in ignition temperature. However, a few studies have closely examined the ignition process of Ni-coated Al particles in air. Shafirovich et al. levitated a micron-sized (15–150  $\mu\text{m}$ ) Ni-coated single Al particle in an electrodynamic levitator and ignited it using  $\text{CO}_2$  laser [26]. In addition, they ignited a free-falling micron-sized (100  $\mu\text{m}$ ) Ni-coated single Al particle in an electro furnace and used the emission data obtained upon ignition of the particle for analysis. The author determined the quantitative characteristic of ignition temperature depending on the concentration of  $\text{O}_2$  as an oxidizer and showed that the ignition delay time decreased with increasing Ni-coating content. In addition, they observed that the ignition temperature increased due to oxygen despite the Ni coating on the Al surface and mentioned the need to understand this phenomenon. Their research on micron-sized particles in air provides an insight into the ignition and burning of Ni-coated Al. However, they had only a very short time to analyze the ignition characteristics due to the experimental environment and a lack of spatial and temporal resolution, which limited their observations of the mechanisms occurring on the particle surface during ignition.

On the other hand, the major early studies on ignition mechanisms explained that Al oxidizes and ignition occurs as the Ni shell structure surrounding the Al breaks under thermal stress [18]. A combustion synthesis experiment to produce Ni aluminide (Ni-Al or  $\text{Ni}_3\text{Al}$ ) using a compact mixture of Ni and Al particles physically mixed together showed that the ignition process starts with the heat of formation of Ni-Al or  $\text{Ni}_3\text{Al}$  by the SHS reaction, which occurs when molten Al flows over the Ni surface [36]. Similarly, a series of ignition experiments on Ni-coated Al powder found that as the Ni coating breaks under thermal expansion, the molten Al leaks out and flows over the Ni surface, and heat supplied by the SHS reaction leads to ignition [31,36]. Shafirovich et al. found that the exothermic intermetallic reactions between liquid Al and solid Ni lowered the ignition temperature, thereby greatly reducing

the ignition delay time of Ni-coated micron-sized single Al particle [26]. This reaction was considered the major factor promoting ignition. They explained the ignition mechanism from a macroscopic perspective, focusing on a millimeter-sized ( $\sim 2.5$  mm) Ni-coated single Al particle in  $\text{CO}_2$  atmosphere. They measured the temperature using a thermocouple and analyzed the images of each ignition stage using a high-speed camera. In addition, the cross-section of the particle was quantitatively analyzed for each ignition stage using the quenching method. Based on their findings, they explained that the ignition mechanism of Ni-coated Al could be developed by exothermic intermetallic reactions and phase transformations of different Ni-Al compounds in  $\text{CO}_2$  atmosphere [24]. Although we conducted research regarding the ignition mechanism in a different oxidizing atmosphere, we were inspired by his experimental approach and analysis [28].

In the present research, a Ni coating was applied to decrease the ignition delay in single particles and the ignition delay effect in air was evaluated. Particles with a size of 2.38 mm were used to ease observation of the detailed structural changes on the Al surface during the entire combustion process (by temperature change) to elucidate the ignition mechanism of Ni-coated Al. The results of this paper are largely divided into two parts. The first part deals with the mechanisms occurring on the particle surface when ignition is performed with a  $\text{CO}_2$  laser on Al coated by electrolysis at a temperature of more than 2000 K. In the second part, Al particles coated by the electroless method and heated in an electric furnace were used to observe thermogravimetry and heat flow (in and out) and provide a fine structural analysis of the particle surface and cross-section. To easily identify the ignition promotion effect of Ni on a single Al particle in the ignition experiments, the coating needs to be thick with Ni in a theoretical mixture ratio that considers the SHS reaction of Ni-Al. However, when Al is used as a fuel for propulsion systems, a thin coating is required because it only serves as a protective film. Therefore, in this research, a relatively thin film was used to help ignite the Al particles and focused on the ignition promotion effect of Ni film on a single Al particle in air.

## 2. Experimental apparatus

We conducted experiments on the combustion of single Ni-coated Al particles in air at room temperature and atmospheric pressure using Al spheres (99% purity, dia. 2.38 mm, Alpha Aesar, USA). These spherical Al particles were preprocessed by dipping in a mixed solution of chromium (VI) oxide ( $\text{CrO}_3$ , Sigma-Aldrich, USA) and phosphoric acid ( $\text{H}_3\text{PO}_4$ , Sigma-Aldrich, USA) at 50°C for 10 min to remove the air-formed oxide film on their surface [37]. To prevent re-oxidation during the Ni coating process, the interval between the processes was minimized. After vacuum drying at 80°C for 10 h, Al particles were stored in a vacuum desiccator before the experiment. The processed Al particles were used approximately 10 times for each condition and then evaluated.

For this research, we used the electro/electroless method to form the coating film on the preconditioned Al surface. There are many ways to coat Ni, but the most popular method is electro-coating. This process is widely used for industrial purposes due to its low cost. Moreover, the hardness of the coating is lower than that produced by other methods, which renders it suitable for inducing successful ignition. Therefore, the first part of the research, which used the electro-coating method, focused on surface observations of the ignition effect of the Ni coating on Al. The Al particles with an electro-coating had a shorter ignition delay time; however, ignition occurred at approximately 2300–2400 K, which is higher than the ignition temperatures reported in previous studies that used other oxidizing conditions or micron-sized particles. Hence, we used the electroless coating method to assess its

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