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# A facile synthesis and efficient thermal oxidation of polytetrafluoroethylene-coated aluminum powders



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## ABSTRACT

Polytetrafluoroethylene (PTFE) films were homogeneously coated onto aluminum powders. The synthesized PTFE-coated aluminum (PTFE/Al) powders showed a microstructure of spherical Al powders covered with PTFE film of a few hundreds of nm in thickness. The PTFE/Al powders showed significantly increased gravimetric energy of  $4.80 \text{ kJ g}^{-1}$  compared to the value of  $0.88 \text{ kJ g}^{-1}$  for pure Al powders during oxidation in a temperature range of 25–1450 °C. It was found that the PTFE/Al interface mixed with oxygen and fluorine atoms provided a vigorous oxidation of the Al powders. These results clearly confirm that the PTFE layer acts as a protective layer and an oxidation helper.

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Aluminum (Al) and its alloyed powders have been utilized for energetic materials such as propellants, solid fuels, and pyrotechnics due to the high thermogravimetric energy release density ( $31 \text{ kJ g}^{-1}$ ) generated caused by the intensive and vigorous oxidation process in an air atmosphere [1–4]. However, practical energetic applications of Al powders are often limited by dense oxide layers which form on their surface [5,6]. These layers tend to prevent the further diffusion of oxygen atoms externally at a low temperature once they form on the surfaces of the Al powders. Thus, it is not easy to achieve a continued exothermic reaction until the oxide materials are removed by melting at an elevated temperature [6].

Therefore, there have been many attempts [7–12] to achieve appropriate combinations of the formation of a surface passivation layer and the enhancement of high reactivity to external oxygen atoms and thus, optimize the energetic properties. Many studies have reported that organic agents as passivation materials should be applied as a coating onto the nano- and micro-scale Al powders [8,12,13]. In particular, epoxide [14] or perfluoroalkyl carboxyl acids [15] has been developed for the passivation of nano-sized Al powders but large-scale production of stable nano-Al powders remains a problem to be solved while also achieving superior reactivity.

Among the many organic passivation materials currently available, fluorocarbons such as polytetrafluoroethylene (PTFE or

Teflon) have been of interest due to their high chemical stability caused by their carbon–fluorine chain and the rapid oxidation rates, which are accelerated by the formation of compounds such as  $\text{AlF}_3$  phase sublimated at an elevated temperature [13,16,17]. That is, it has been reported that the formation of aluminum or carbon fluorides helps to create a new surface on Al powders through their evaporation during the oxidation process. The newly formed surface can produce a large-area reactive plane. Recently, Sippel et al. [8] showed Al agglomeration using Al/PTFE mixtures for the propellant, with the mechanism of the combustion of mixed Al/PTFE elucidated. However, there have been few results pertaining to the homogenous coating of PTFE materials on the surface of Al powders thus far, despite the high likelihood that coated PTFE can be efficiently utilized as an oxidation helper for Al powders compared to mechanically mixed substances. Once PTFE is coated onto a pure Al powder surface with a minimized oxide layer, the reactivity of the powders can be consistently improved and high chemical and thermal stability of the Al powders by protective coating can be expected.

Hence, in this study, a coating process of PTFE nanoparticles to create film on spherical Al powders is introduced. The role of the coated PTFE film in the thermal oxidation of pure Al powders is then studied through an analysis of the interfacial structure between the PTFE and the Al powders.

Commercial PTFE nanoparticles (purchased from Dyneon Co. Ltd.) approximately 200 nm in size dispersed in an aqueous solution were used as the coating materials. Spherical Al powders 45  $\mu\text{m}$  in size on average synthesized by a gas-atomization process

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were used as the energetic materials. Before the coating with the PTFE nanoparticles, the oxide layer present on the Al powder surface was removed using a pH-controlled sodium hydroxide solution. The  $\text{Al}_2\text{O}_3$  film was dissolved in a solution in the form of an Al-hydroxide-forming hydrogen gas [9]. The generated hydrogen bubbles simultaneously broke the  $\text{Al}_2\text{O}_3$  film [6]. The Al powders after oxide removal were mixed and stirred with PTFE nanoparticles, after which the PTFE nanoparticles were attached onto the pure Al surface at an elevated temperature due to the high affinity between aluminum and fluorine atoms. The PTFE nanoparticle-attached Al powders were filtered and dried at 80 °C for 1 h under an Ar atmosphere. Finally, a heat treatment of the PTFE nanoparticle-coated Al powders was applied at 300 °C and maintained for 1 h in order to coat the PTFE as a layer. The microstructures of the synthesized PTFE-coated Al powders were characterized by scanning electron microscopy (SEM, JEOL, JSM-5800). The distribution of the PTFE and Al atoms were evaluated by energy-dispersive X-ray spectroscopy (EDS) equipped in the SEM. A thermogravimetric analysis (TGA, SDT Model no. Q600) and a differential scanning calorimeter (DSC, SDT Model no. Q600) were used to compare both the thermal oxidation results and the exothermic reaction behavior between the PTFE-coated Al powders and the pure Al powders. The thermal analysis was performed in an air atmosphere for thermal oxidation at a heating rate of 5 °C/min. The microstructure of the PTFE/Al interface was characterized by field emission transmission electron microscopy (FE-TEM, JEM-ARM200F). The formation of PTFE was confirmed by means of Fourier transformed infrared spectroscopy (FT-IR).

Fig. 1(a) schematically illustrates the key processes used to synthesize the PTFE-coated Al powders. The Al powders are initially dispersed in an aqueous solution including NaOH in order to remove the surface oxide. As the pH reaches a level of 9–10,  $\text{Al}_2\text{O}_3$  can be dissolved in the form of Al hydroxide materials. The PTFE

nanoparticles in the solution are added to the solution of Al powders with the minimized oxide layer. The attached PTFE nanoparticles are molten and form into a film on the pure Al powders due to the heat treatment. Fig. 1(b) shows a surface SEM image of the PTFE-coated Al powders, illustrating their spherical shape. The EDS mapping results in Fig. 1(c) show that fluorine atoms are homogeneously distributed with the Al powders. Two characteristic peaks are observed at 1150 and 1250  $\text{cm}^{-1}$  in the FT-IR graph of PTFE as shown in Fig. 1(d) and (e). These peaks are similarly revealed for PTFE/Al powders at same wave number. In addition, bonding between carbon and oxygen atoms caused by breaking of PTFE chain is observed near 1600  $\text{cm}^{-1}$ . This indicates that the coated materials are PTFE and that they survived even after a heat treatment at an elevated temperature.

Fig. 2(a) displays a TEM image showing the PTFE/Al interface at a low magnification. This figure reveals that the thickness of the PTFE layer is approximately a few hundred nm. The SAED patterns in the inset can be identified as the [112] zone axis of the FCC Al phase. The interfacial region marked by the white square box in Fig. 2(a) was enlarged. The EDS results displaying the distribution of the oxygen, fluorine, aluminum and carbon atoms are shown in Fig. 2(b). A highly concentrated region of oxygen, carbon and fluorine atoms was found near the PTFE/Al interface as schematically illustrated in Fig. 2(c). This indicates that carbon and fluorine atoms can be readily introduced into Al oxide film or on the surface of Al powders where the oxide layer is peeled off.

Fig. 3(a) shows a comparison of the TGA results between the PTFE/Al and the pure Al powders in the temperature range of 25–1450 °C. There is no significant change in the weight gain of the pure Al except in the region past 1000 °C, which may correspond to the oxidation of Al. Compared to the pure Al, the PTFE/Al powders show a region with 5% reduction in the weight gain from 420 °C to 900 °C (Phase II); this likely occurred because the

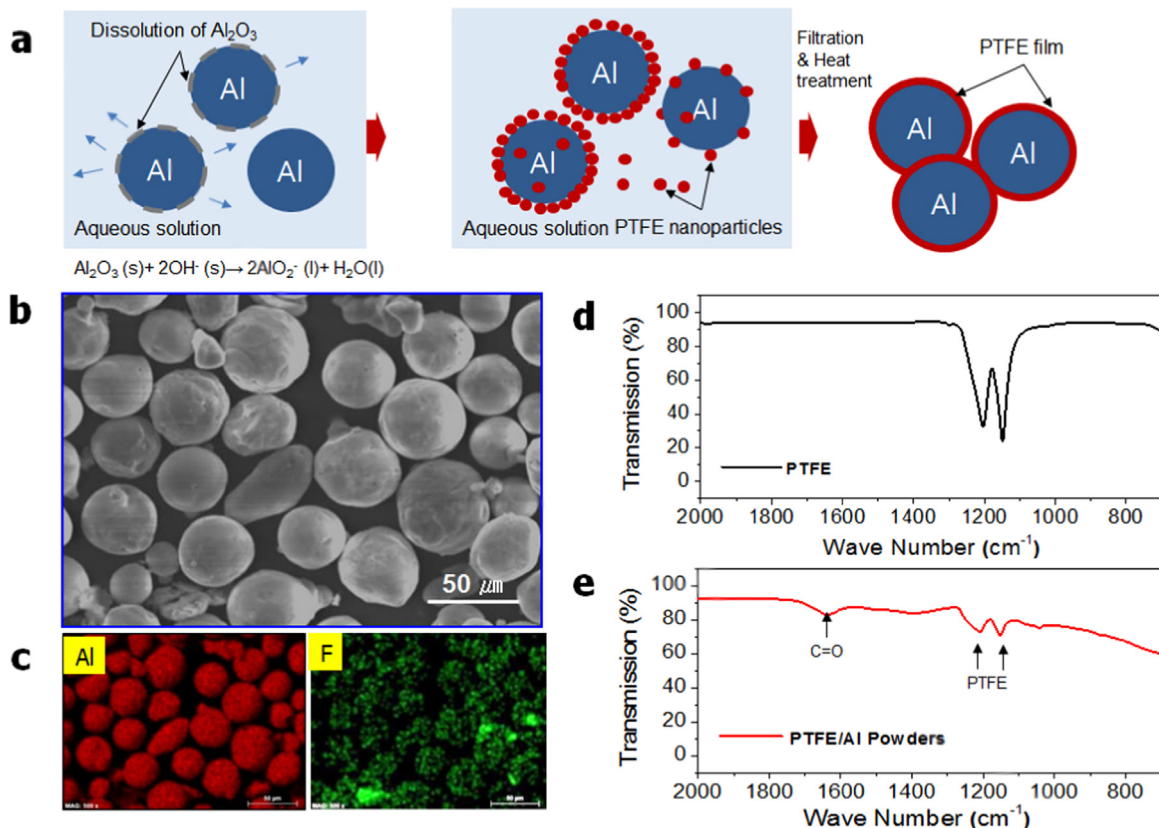


Fig. 1. (a) Schematic illustration showing the synthetic process of the PTFE/Al powders, (b) a SEM image of the synthesized PTFE/Al powders, (c) EDS results showing the distributions of aluminum and fluorine atoms, and (d) FT-IR spectral analysis of the PTFE/Al powders compared with PTFE.

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