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Original Research Paper

Propellant grade ultrafine aluminum powder by RF induction plasma

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ABSTRACT

A continuous process of synthesizing ultrafine aluminum powder (UFAP) having high yield rate using RF induction plasma is reported in the current work. The processing parameters (powder injection probe position; powder feed rate; flow rates of gases for plasma forming, quenching and passivation) were varied systematically to evaluate their influence on the size and distribution, and metallic Al content of the synthesized powder. The UFAP was characterized using X–ray diffraction and, scanning and transmission electron microscopes. The metallic aluminum content of UFAP was evaluated using hydrogen gas evolution technique and its thermal behavior was studied using simultaneous thermo–gravimetric and differential scanning calorimetry under static air. UFAP with an average particle size varying between 220 and 400 nm was obtained under different processing conditions and a maximum metallic Al content of 89.2% was obtained at a high powder feed rate. Among the parameters studied, increased powder feed rate, plasma forming gas flow rate and the position of the powder injection probe had a significant effect on the particle size, distribution and the metallic Al content of the synthesized UFAP.

1. Introduction

Aluminum powder is commonly used in a wide variety of applications like rocket propellant additives [1,2], thermite mixtures [3], paints [4] and hydrogen generation [5]. The use of micron sized Al powder as propellant additive leads to incomplete combustion, high ignition temperature and ignition delay [6]. This results in deposition of unburnt or partially burnt metal deposits on the combustion chamber [7,8], thereby reducing the thrust. The loss in thrust also arises due to obstruction to the flowing gases by particle drag [7]. The decrease in particle diameter by a factor of 10 reduces the burning time by a factor of 100 according to D^2 law [6]. Hence, research is focused on developing nano/ultrafine Al powder. Complete combustion circumvents the loss in thrust encountered while using nano/ultrafine Al powder. However, decreasing the particle size to nano regime increases the surface aluminum oxide content to significant limits thus reducing the available active metallic Al content [9]. The decreased metallic Al content decreases the available oxidation enthalpy, which affects the propellant performance, namely, the ideal specific impulse [1]. Hence, UFAP of size 100 nm^{-1} μm is a preferred propellant additive as it promotes complete combustion.

Nano/UFAP can be synthesized by a number of techniques like electric explosion of wire [10,11], mechanical milling [12], chemical synthesis [13], inert gas condensation [14,15] and H_2 plasma evaporation [16]. Recently, radio frequency induction plasma (RFIP) process has been successfully used to synthesize various metal [17,18], alloy [19,20], oxide [21,22] and nitride [23] powders. This process offers several advantages, namely: (a) larger plasma volume coupled with lower velocity compared to DC plasma, with longer residence time inside the discharge zone; (b) different types of feedstock materials (solid/liquid/gaseous) can be used; (c) contamination of the synthesized powder is low since RFIP setup has no electrodes; (d) central injection of feed stock allows in–fight evaporation of raw materials even with highest boiling points; (e) provision to operate under different atmospheres, thus permitting synthesis of various powders; (f) flexibility to change feed rate of powder during the process, independent of RFIP parameters; and (g) continuous process to synthesize nano/ultrafine powder with high productivity of the order of $\sim 0.5^{-1}$ kg/hr.

Table 1 lists some of the commonly used techniques for the synthesis of ultrafine Al powders. Though DC plasma and gas condensation techniques produce powders in terms of kilogram quantity, they suffer from short term or batch type process, respectively. However, RFIP has got the twin advantages of higher productivity as well as the ability to operate under continuous mode.

RFIP operates at temperatures above 10^4 K and is quenched (10^5–10^6 K/s) at the tail of the plasma plume [30]. The process

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Table 1
Some of the commonly used techniques for the synthesis of ultrafine Al powders.

Method	Quoted size, nm	Production rate	Reference
Electro–explosion of wire	50–500	100's g/hr	[24]
DC plasma torch	50–150	2 kg/hr short run	[25]
Gas condensation	20–200	kg/hr/reactor, batch process	[26]
Laser induction complex heating apparatus	30–100	15 g/hr	[27]
Transferred arc thermal plasma reactor	50–250	50 g/hr	[28]
Arc plasma spray	30–80	120 g/hr	[29]
Radio frequency induction plasma	50–200	kg/hr, continuous process	This study

essentially encompasses induction heating followed by inert gas condensation to yield ultrafine particles. However, the heating process of injected metal precursors in RFIP is significantly dependent on various operational parameters, such as, plasma power, and precursor feed rate and size [31]. The effect of processing parameters on Cu particle size was reported by Kobayashi et al. [18]. The influence of quenching on the powder size and distribution for alumina and silica powders are also reported [22,32]. Leparoux et al. [33] have investigated the ability of RFIP to control the particle size and stoichiometry of TiC_xN_{1-x} nano powder.

Thus, from the limited studies reported, it is clear that the processing parameters of induction plasma will have a direct impact on the particle size and distribution of the synthesized powder. However, no such information is available for aluminum, a material which has got great demand in applications such as propellants [1–3]. Therefore, the primary objective of this work was to understand the parametric influence of the induction plasma processing on particle size and distribution of the synthesized UFAP, while the other objective was to optimize the condition, which yields the highest metallic Al content.

2. Experimental details

2.1. Induction plasma processing for UFAP

The UFAP was processed in an RFIP set up details of which are given elsewhere [22,31–35]. However, it is briefly described here. The RFIP consists of three major parts, *viz.* the reaction chamber, quench part and powder collection filter. A Tekna plasma torch (Tekna Plasma Systems, Inc., Quebec, Canada) operated at 2–5 MHz frequency with a maximum plate power of 60 kW was used. The torch is composed of two coaxial quartz tubes of 105 mm length, the inside quartz tube having the internal diameter of 50 mm. The central gas (also known as the plasma forming gas) stabilizes the plasma, and the sheath gas cools down the torch wall from high temperature of the discharge. The feedstock is fed axially into the plasma plume through a water cooled probe. The internal diameter, external diameter and length of the powder injection probe are 4.7 mm, 9.5 mm and 130 mm, respectively. The high enthalpy plasma melts and vaporizes the precursor powder almost instantly, which is subsequently quenched as it comes out of the plasma chamber. The filtration chamber consists of porous SS316L filters, which effectively separates the solid powder from the gaseous products.

2.2. RFIP process parameters

The different process parameters such as powder injection probe position, powder feed rate (PFR), and flow rate of plasma forming gas, quench gas and passivation gas modifies the synthesis environment and consequently influences the particle size and distribution of the synthesized powder. In this study, argon was used as carrier, plasma forming (central) and quench gases and hydrogen was used as sheath gas in addition to argon. The purpose of using hydrogen gas in addition to argon as sheath gas is to protect the plasma confinement tube from damage by the high temperature plasma. However, only argon and not hydrogen gas was used as powder carrier gas, as hydrogen in the carrier gas will substantially cool down the plasma at the injection point. Fig. 1 depicts the schematic representation of RF induction plasma equipment showing the powder carrier gas, plasma forming gas, sheath gas and quenching gas.

The parameters that were varied to study their effects on the synthesized UFAP average particle size, distribution and the metallic Al content are listed in Table 2.

The lower and upper limit values of the process variables were selected in the following manner:

- Passivation gas flow rate (PGFR): The higher level of PGFR was fixed at 0.3 slpm of oxygen to ensure complete passivation to avoid powder catching fire upon exposure to air. The lower limit of 0.1 slpm was fixed as the powder after passivation with 0.1 slpm oxygen was found to be slightly warm upon exposure to air. It is envisaged that a PGFR lower than 0.1 slpm would leave the powder insufficiently passivated, possibly leading to fire related accidents.
- The position of powder injection probe was chosen such that it does not overheat and damage the injection probe.
- The maximum PFR was limited to 40 g/min so as not to choke the stainless steel powder injection probe. When a feed rate of 50 g/min was employed, it blocked the feeding probe after

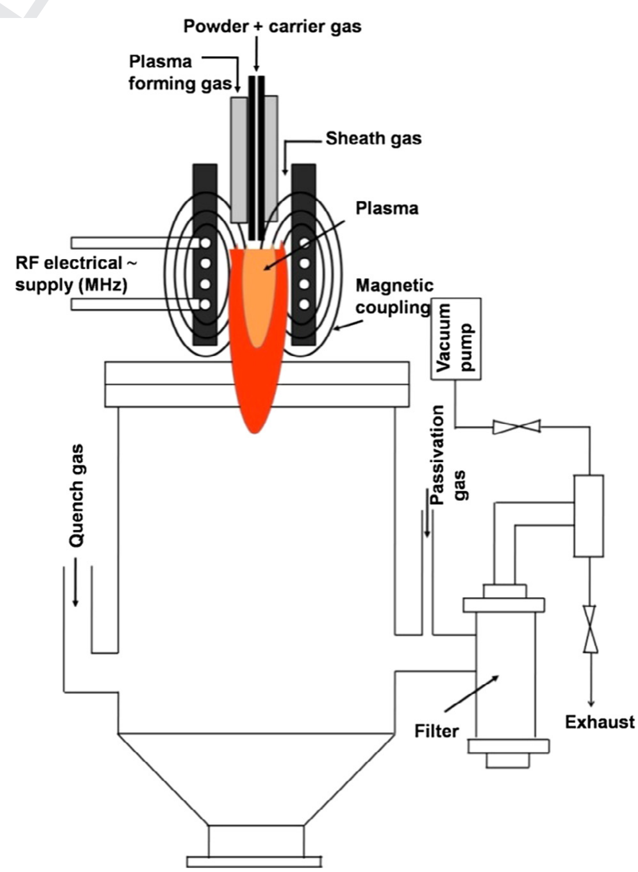

Fig. 1. Schematic representation of RF induction plasma equipment showing the powder carrier gas, plasma forming gas, sheath gas and quenching gas.

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