



# Cold welding behavior of fine bare aluminum powders prepared by new low oxygen induction thermal plasma system

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## ABSTRACT

A low oxygen induction thermal plasma (LO-ITP) system was developed and a cold-welding behavior of bare fine metallic aluminum powders prepared by the system developed here was observed. This behavior is suitable for a metal precursor for metal matrix composites (MMC) since fine and bare metal particles may contribute to obtaining a well-dispersed state and a high relative density respectively, which lead to the enhancement of the reinforcement of MMC properties. The electric conductivity of the aluminum green pellet compacted at 200 MPa reached  $2.9 \times 10^7$  S/m, which is comparable to that of bulk aluminum, indicating that the cold welding was taken place since the surface contamination layer such as an oxide may be negligibly thin. Therefore, the powder obtained in this work is expected to be useful in enhancing the reinforcement of MMC properties.

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## 1. Introduction

Recently, interest in metal matrix composites (MMC) has been stimulated, in part, by their potential for reinforcement of physical properties such as hardness, wear resistance, the friction coefficient and thermal and electrical conductivity. In particular, property reinforcement materials such as carbon nanotubes (CNT) [1], graphene [2] and boron nitride nanotubes (BNNT) [3] have received considerable attention owing to their remarkable properties of high strength, high thermal conductivity and high electrical conductivity, realizing that higher properties may be obtained in MMCs containing these materials than in the simple metals. Although uniform dispersion in the metal matrix is effective for enhancing property reinforcement of Al-CNT MMC [4,5] and Al-graphene MMC [6–9], for example, it is nevertheless a challenging issue since the size and aspect ratio of the metals and the reinforcement materials are substantially different, and there are also strong Van der Waals forces between the reinforcement materials, which tend to cause aggregation in the metal matrix [7].

One successful process for obtaining a well-dispersed MMC is a bottom-up process such as electrodeposition, as nucleation occurs slowly on the surface of the aligned reinforcement material and the

nuclei then grow and fill the matrix of the reinforcement material. As a result, a CNT-Cu MMC prepared by an electrodeposition technique showed large enhancement of the current carrying capacity because the polycrystalline Cu was tightly bound with long, intertwined, well-dispersed CNTs. This MMC has been obtained successfully at both the lab scale [10] and in a large-scale process [11]. However, this method is only used with metals that can be reduced by hydrogen, since a surface contamination layer, especially an oxide layer, which was formed when deposition was performed with an aqueous solution, should be removed. Thus, the problem is how to realize a similar well-dispersed state with metals that cannot be reduced and cleaned by hydrogen, such as aluminum and titanium. In order to realize a well-dispersed state, a particle with a size smaller than or at least comparable to that of the reinforcement material is suitable when mixing these metals with reinforcement materials. Moreover, this fine metal particles should be bare (i.e. without contamination such as an oxide), since the oxide layer was prevented to the direct contact between the metallic Al particle [12], which is challenging issue for easily oxidizable materials because the specific surface area increases as the particle size decreases. Once the bare surface is obtained, the strong direct contact of particle-to-particle and particle-to-reinforcement materials is expected, realizing a cold welding under a certain pressure.

Aluminum is known to be one of the most easily oxidizable metals among those used as the metal matrix of MMC so far.

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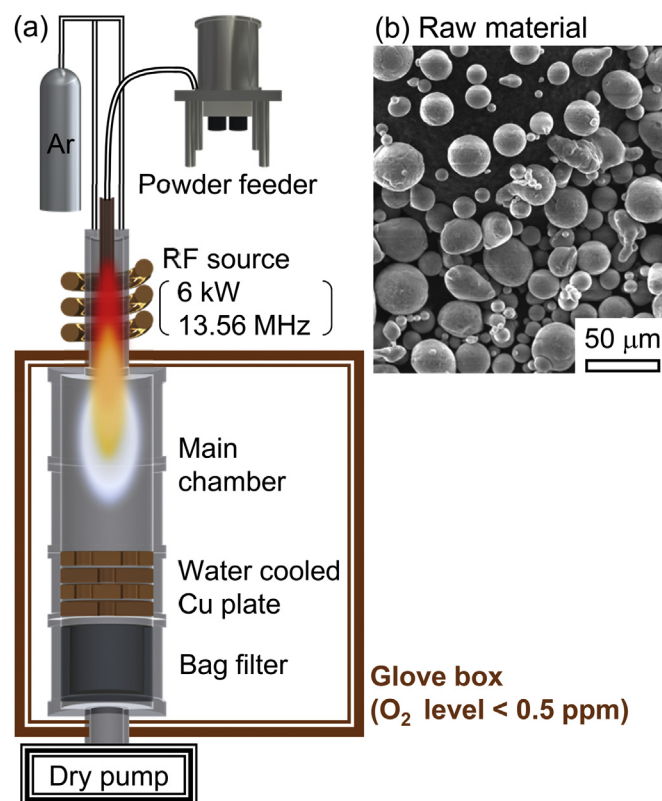
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Various techniques for preparing nano-sized aluminum particles have been reported [13], including gas evaporation [14], arc discharge [15], pulsed laser ablation [16], electro explosion and chemical solution methods [17]. Among these techniques, we focused on the induction thermal plasma (ITP) technique as a gas evaporation process with the remarkable advantages of a high production rate, controllability of the particle size (ideally mono-dispersed) [18,19] and an inherently contamination-free process, as no electrode is used. Here, an ITP process will be introduced briefly. The ITP is produced by high frequency electromagnetic induction, and its temperature reaches as much as 10 000 K. When a raw powder material is injected into such a high temperature plasma, the powder is vaporized and fine particles are produced through a process of nucleation, condensation and coagulation with a high cooling rate of  $10^3$ – $10^6$  K/s. To our knowledge, Karthik et al. was the first to report the preparation of a fine metallic aluminum powder by an ITP route, and studied the significant parameters which affected the particle size and distribution and the metallic aluminum content [20]. However, they did not take care to prevent oxidation because the powders were collected after passivation by oxygen. In order to obtain a “bare” fine powder, in this work, we developed a low oxygen induction thermal plasma (LO-ITP) system to inhibit oxidation, and focused on the preparation of fine bare aluminum powders as a demonstration.

## 2. Experimental detail

A schematic illustration of the LO-ITP is shown in Fig. 1(a). A TP-40020NPS (JEOL Co., Ltd.) was used as the ITP process itself, and a TP-99010FDR (JEOL Co., Ltd.) was used as the powder feeding system. The power of the RF generator was 6 kW with a frequency of 13.56 MHz. The starting material was the coarse metallic aluminum powder (particle size  $<45 \mu\text{m}$ , purity 99.99%, oxygen level 0.095 wt %, Kojundo Chemical Lab. Co., Ltd., Japan) shown in Fig. 1(b). As a feature of this system, we also constructed a glove box which was capable of maintaining an oxygen level of  $<0.5$  ppm so as to minimize oxidation as far as possible when collecting the processed powder. In the thermal plasma process, first, Ar (G1 grade, oxygen level of less than 0.1 ppm) was introduced in the main chamber up to a process pressure,  $P_{\text{proc}}$ , of 40, 70 or 90 kPa. The raw aluminum powder was then introduced from the top of the plasma torch at a feed rate of up to 0.3 g/min. Here, Ar were used as the plasma and carrier gas, and their flow rates were 35 and 3 L/min., respectively. The powder was collected from the wall of the main chamber and the Cu plates and compressed at 100, 200 or 500 MPa at room temperature by using an oil hydraulic press in the glove box. In the following, the samples are denoted as [ $P_{\text{proc}}$ , compression pressure]. The diameter and thickness of the green compacts were 6 mm and 0.3–0.5 mm, respectively.

The phase was confirmed by XRD (PANalytical, Empyrean,  $\text{Co-K}\alpha$ ). The particle morphology and size distribution were estimated from images taken with a scanning electron microscope (SEM; JEOL, JSM-7800F). The particle size was defined as the mean value of  $e^{\mu+\theta^2/2}$ , where  $\mu$  and  $\theta$  are parameters obtained by fitting to the log-normal distribution,  $1/\sqrt{2\pi}\theta \exp(-(\ln x - \mu)^2/2\theta^2)$ . The standard deviation  $\sigma$  defined as  $e^{2\mu+\theta^2}(e^{\theta^2} - 1)$  was also estimated. The electric conductivity of the green compacts was measured by the four probe method using a Loresta-GX MCP-T700 (Mitsubishi Chemical Analytech Co., Ltd.) at room temperature. The relative density of each green pellet was estimated by the Archimedes method. The bulk aluminum density of  $2.7 \text{ g/cm}^3$  was used. The oxygen level of the powders was measured by EMGA-620W (HORIBA, Ltd) without atmospheric exposure.



**Fig. 1.** (a) Schematic illustration of experimental set up, which consists mainly of four parts: RF torch, chamber, powder feeder and glove box. The thermal plasma processed powder was collected from the walls of the main chamber. A glove box which maintains an oxygen level lower than 0.5 ppm was constructed for powder collection in order to inhibit oxidation as much as possible. Some pellets with sizes of  $\phi 6 \times 0.3$ – $0.5$  mm were obtained for evaluation of electrical conductivity by using an oil hydraulic press, which was placed in the glove box. (b) SEM image of Al raw material. The particle size of the coarse metallic aluminum powder was less than  $45 \mu\text{m}$ .

## 3. Results and discussion

Fig. 2 shows SEM images of the fine aluminum powders processed at (a) 40 kPa, (b) 70 kPa and (c) 90 kPa, respectively. The shape of all particles was spherical. A histogram was constructed based on these images, as shown in Fig. 2(d). Here, the total particle number  $N$  of observations is 500 for each powder and bin is 25 nm. The mean particle sizes of 79 nm ( $\sigma = 31$  nm), 239 nm ( $\sigma = 92$  nm) and 134 nm ( $\sigma = 61$  nm) were estimated from the SEM images shown in Fig. 2(a), (b) and (c), respectively, by fitting to the log-normal distribution. The mean particle size and  $\sigma$  increased with increasing  $P_{\text{proc}}$  from 40 kPa, reached a maximum at around  $P_{\text{proc}}$  of 70 kPa, and then decreased with a further increase in  $P_{\text{proc}}$  to 90 kPa. This tendency may be understood from the following trade-off relationship. Smaller particles processed under higher  $P_{\text{proc}}$  are expected to be obtained since higher  $P_{\text{proc}}$  leads to a higher quenching rate due to the higher thermal conductivity of the Ar gas in the plasma. The particle size may increase at higher  $P_{\text{proc}}$  due to the higher density of the element in the temperature region of coagulation and condensation, realizing a shorter mean free path. Consequently, the particle size displays a local maximum between the  $P_{\text{proc}}$  of 40–90 kPa for aluminum, which is partly consistent with the previously reported results [19]. It should be noted that this behavior may be strongly related to the experimental setup, that is, the chamber size and shape, input power and raw powder feeding rate [18]. The XRD spectra show a single phase of aluminum

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