Journal of Materials Processing Tech. 252 (2018) 559–566

Contents lists available at ScienceDirect

Journal of Materials Processing Tech.

journal homepage: www.elsevier.com/locate/jmatprotec

Research Paper

# Plasma-assisted preparation and characterization of spherical stainless steel powders

Hailong Zhu[a,*], Honghui Tong[b], Fazhan Yang[b], Changming Cheng[b]

[a] College of Physics and Electronics Engineering, Shanxi University, Wucheng Road 92, Taiyuan 030006, China
[b] Southwestern Institute of Physics, Chengdu 610041, China

ARTICLE INFO

*Keywords:*
Spherical stainless steel powder
Thermal plasma
Characterization
Additive manufacturing

ABSTRACT

A study of the plasma-assisted rapid melting-spheroidizing-solidification process was carried out for the preparation of micron-sized spherical stainless steel powders. The melting behavior of the in-flight particles was investigated through the particles temperature monitoring with DPV-2000. The morphology, powder size distribution, phase and pore structure, densities and flowability of the raw powder and the as-prepared spherical stainless steel powder were characterized and analyzed comparatively. It was demonstrated that most powder particles were melted into droplets at the designated experimental conditions, whereas the as-prepared stainless steel powder demonstrated excellent sphericity, flowability and monodispersity. A fraction of nanoparticles and hollow powders were produced along with the spherical powder during the plasma process. The apparent density, the tap density, the flowability and the Brunauer–Emmett–Teller surface area of the as-prepared spherical powder were 3.50 g $cm^{-3}$, 5.20 g $cm^{-3}$, 15.2 s (50 g)$^{-1}$, 1.59 $m^2$ $g^{-1}$, respectively, whereas the compositions and powder size distribution had almost no change after plasma treatment. The as-prepared spherical stainless steel powder could be utilized as a candidate powder for additive manufacturing.

## 1. Introduction

The micron-sized spherical powders with a series of unique advantages such as good flowability, high apparent density and tap density, as well as high specific surface area, have been of particular interest to researchers due to these advantages and potential applications in electronic packaging (Ai et al., 2011), fuel cells (Maric et al., 2000), thermal spraying (Qian et al., 2009), metal injection molding (Walther et al., 2014) and additive manufacturing (AM) (Sun et al., 2016). Except the aforementioned advantages, the micron-sized spherical stainless steel powder (SSP) has the specific characteristics of low melting point, good toughness, high corrosion resistance, high density and low cost, being considered as one of the best candidates powders for AM system (Slotwinski et al., 2014). The study on preparation and characterization of micron-sized spherical SSP were executed successively, rapidly becoming a certain research hotspot in the AM field.

The spherical powder could be prepared through two main approaches: (i) the mechanical process approach, where the raw material is crushed into irregular fine powders by extrusion and impact, further grinded into spherical powders, however, the chemical composition of the powders have almost no change in the entire process. Also (ii) the physicochemical approach exists, where the condensation state or the chemical composition of the raw materials, were changed through a physical action or chemical reaction resulting in the production of spherical powders. As a typical physicochemical method, the thermal plasma was widely utilized in the preparation of spherical powders due to the corresponding unique advantages such as high temperature, high enthalpy, chemical activity and fast quenching rate (Mostaghimi and Boulos, 2015). Once the raw powder (*solid phase*), particularly, for the low melting point powder (stainless steel powder with a melting point of 1516 °C), was injected into the plasma body, it was heated and melted down immediately formed the droplet (*liquid phase*), due to the high temperature of the thermal plasma, the melted droplet formed a spherical droplet due to surface tension. Subsequently the melted droplets would solidify into a spherical powder (*solid phase*), due to rapid quenching when the former spurt out of the plasma, as presented in Fig. 1. During the plasma processing, since the powder particle sustained an incredibly short duration (approximately several milliseconds) in the thermal plasma. This method can be considered as a rapid melting and solidification process of powder particles and one-step-preparation, demonstrating the distinctive advantages in the preparation of spherical powders. Compared to other preparation methods such as chemical reaction method (Zhao et al., 2009) and flame spheroidization process (Jin et al., 2010), the other highlight advantage

* Corresponding author.
*E-mail address:* zhuhl@sxu.edu.cn (H. Zhu).

http://dx.doi.org/10.1016/j.jmatprotec.2017.10.010
Received 2 May 2017; Received in revised form 30 August 2017; Accepted 4 October 2017
Available online 06 October 2017
0924-0136/ 

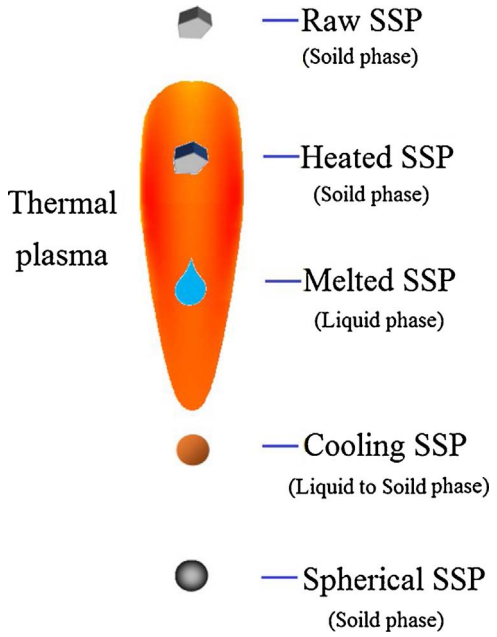


**Fig. 1.** Schematic illustration of spherical SSP preparation.

of this method was an easier operation and without secondary pollution introduction, such as waste water and toxic gases. Therefore, various powders, such as the refractory metal and ceramic powders were prepared in succession based on the thermal plasma process for various research backgrounds and application situations. Sun et al. (2007) presented the preparation of rare earth oxide powders by thermal plasma, where the powders were doped into multi-layer ceramic capacitors to improve the lifetime. Kobayashi et al. (2008) studied the synthesis mechanism of spherical submicron copper powder, which can be applied to the conductive fillers. The authors' research group conducted a high amount of work on the refractory metal (Zhu et al., 2016) and ceramic powders (Zhu et al., 2017) preparation, which were used as fusion materials. However, the preparation of spherical SSP by thermal plasma, have still rarely been reported (Yang et al., 2015).

The thermal plasma was proved to be effective in various spherical powder preparations. A further study should be conducted because the powder particle sustained complex effects of both heat and flow during the thermal plasma processing. The surface feature and internal structure of the powder changed, highly affecting the product final properties. This paper was aimed at the preparation of spherical SSP for the corresponding potential application in the AM field, and the properties change of the SSP during this process. In order to determine the physicochemical properties of the as-prepared powder, certain characterization techniques were performed on the morphology, chemical compositions, flowability, density variations, porosity and size distributions, by the scanning electron microscope (SEM), the X-ray diffraction (XRD), the Hall flowmeter, the density tester (DT) and the Brunauer–Emmett–Teller (BET) surface area analysis, respectively.

## 2. Experimental

### 2.1. Experimental set-up and methodology

In Fig. 2, a custom-made equipment of powder plasma treatment consisting of an electric tube radio frequency (RF) power source system, a gas supply system, an exhaust system, a plasma torch, a power feed and a collection system, was utilized for the spherical SSP preparation. The function of each equipment part was introduced in details (Zhu et al., 2017). A commercially available SSP (Lide powder material Co., Ltd, Shijiazhuang, China) with a random edged morphology manufactured by gas atomization was utilized as the raw powder.

During a typical experimental procedure, firstly, a soft vacuum of approximately 40 kPa was created by the vacuum pump until the chamber was emptied from air as the maximum possible prior to the plasma ignition. Secondly, the stable RF thermal plasma was ignited and maintained by applying an RF alternating current oscillating to the coil of the plasma torch. Thirdly, the raw powder was injected uninterruptedly into the plasma high temperature region by an argon carried gas. The raw SSP sustained rapid melting-spheroidizing-solidification in the RF thermal plasma system. A DPV-2000 monitoring instrument (TECNAR Automation, St-Bruno, QC, Canada) fixed at the end of the plasma flame (at 80 mm below the plasma torch spout), was employed for the in-flight particle temperature monitoring and the further SSP melting state estimation. Finally, the formed spherical SSP was collected in the collection port. The as-obtained samples were characterized and analyzed with various instruments. Table 1 presents the utilized operating parameters in the experiment for the SSP preparation. The optimized operating parameters included two parts. The first part refers to the operation parameters of power supply, chamber gas pressure and gas flow rate, which are used to produce and control the plasma. Through the operation parameters adjustments, the plasma reached a stable state, which was characterized by the stable anodic current. The powder feed rate was another determining parameter for the actual plasma processing. The low feed rate would result in higher powder spheroidization rate. In contrast, the low feed rate was extremely unfavorable for the yield rate of the SSP powder. To achieve an optimum balance between the spheroidization rate and yield rate, a feed rate of 50 g/min was selected as the appropriate level in this work.

### 2.2. Characterization methods

The field emission scanning electron microscopy (FE-SEM, Model S-4800, Hitachi, Tokyo, Japan) was utilized in the sample morphologies characterization at an accelerating voltage of 5 kV and linked with an EDS X-Max Oxford spectrometer. The EDS mapping of the samples was performed for the SSP surface composites to be determined.

The pore and phase structures of the raw powder and the as-prepared powder structures were characterized by the Brunauer–Emmett–Teller (BET) surface area analysis and the X-ray Diffraction (XRD) spectroscopy analysis. The specific surface areas of the samples were measured by nitrogen adsorption–desorption on the ASAP2020 instrument. The sample masses of approximately 0.96 g were initially degassed at 363 K (90 °C) for 30 min and subsequently at 573 K (300 °C) for 4 h under evacuation until a pressure of below 1 Pa was achieved. Following cooling down, the sample was re-weighed and analyzed by $N_2$ gas physisorption under liquid nitrogen at 77 K (–196 °C) from an initial pressure of approximately 0.2 Pa to an ambient pressure. The pore size distributions of the samples were calculated by the Barrett–Joyner–Halenda (BJH) method. Also, the average pore sizes were obtained from the peak positions of the distribution curves. The total pore volume was accumulated at a relative pressure of $P/P_0 = 0.99$. The phase structure of the powder samples was characterized by X-ray diffraction (XRD, X'pert Pro MPD, Philips, Netherlands) with Cu $K_\alpha$ radiation in the 0–90° 2θ range. The JCPDS-cards were utilized in the identity determination of any existing phase and the corresponding phase structure.

The sample powder size distribution was measured by a wet laser particle size analyzer (Winner2005A, Jinan, China) in a liquid phase (water was utilized as the dispersant medium).

The sample apparent density values were determined by the standard cup and funnel method, whereas the sample tap density was measured by a tap density tester (FT-100A, Ningbo, China) and the sample flowability was investigated by a calibrated funnel (the Hall flowmeter).