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Modification of cryomilling process to tailor geometrical characteristics of nanostructured Al powder for cold spraying

Bo Feng [a,b], Naeem ul Haq Tariq [a,b,d], Jiqiang Wang [a,*], Hao Du [a], Xusheng Zang [a,c], Tianying Xiong [a,*]

[a] Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, PR China
[b] University of Chinese Academy of Science, No. 19 (A) Yuquan Road, Shijingshan District, Beijing 100049, PR China
[c] School of Materials Science and Engineering, University of Science and Technology of China, Shenyang 110016, PR China
[d] Department of Metallurgy and Materials Engineering, Pakistan Institute of Engineering and Applied Sciences, Nilore, Islamabad, Pakistan

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ABSTRACT

Cryomilling is the most commonly used method to fabricate ultra-fine grained (UFG) or nanostructured metallic powder. However, it has relatively low control over powder's geometric characteristics, particularly for the powders having FCC crystal structure. This shortcoming makes traditional cryomilling process unattractive to produce UFG/nanostructured feedstock for cold spraying process. In the present work, a modified pot vibration milling process was successfully employed to produce nanostructured powder of pure aluminum at 100 K. The geometrical characteristics of Al powder was controlled by using various combinations of the milling media (i.e. steel balls) of different sizes. The results indicate that with increase in milling time, the initial grain size of Al (~1–5 μm) was progressively refined to ~30–50 nm. Moreover, lamellar powder morphology was obtained with the addition of process control agent (PCA) while equiaxed polygonal structure was evolved without using PCA. It was also revealed that, in the absence of PCA, the particle size distribution was mainly controlled by the interstice size distribution of the milling media.

1. Introduction

Nano technology is the foundation of modern material science and engineering. In early 1980s, H. Gleiter first proposed the concept of nanostructured material [1]. Materials at nanometer length scale may exhibit physical properties that are entirely different from the characteristics of their bulk state [2]. In bulk nanostructured materials, the grain refinement usually leads to superior mechanical and physical properties as compared to their coarse-grained counterparts [3–5].

In powder metallurgy, high-energy milling of the powder followed by a consolidation process is the most commonly used method to prepare nanostructured materials. The fabrication of nanostructured powder by high-energy milling has some advantages over other powder processing methods, e.g. high output, flexibility of the process and simplicity of the equipment. For the past three decades, this area has attained a great attention of the researchers [6,7]. However, many studies show that due to the structural features of FCC materials, it is very difficult to form nano-crystalline structure at room temperature due to undesired recovery (RV) and recrystallization (RX) phenomena.

Cryomilling is a mechanical attrition technique in which powders are milled at very low temperature, which usually depend on the liquid nitrogen (LN2) refrigeration process. At cryogenic temperature (~100 K) RV and RX processes become extremely sluggish while dispersed metal nitrides are formed in the milled powder. As a result, nano-crystallization of FCC powder material is facilitated [8,9]. Meanwhile, the powder fabricated by this method has relatively high strength and high thermal stability [9]. In the past, this technique was used to produce nanocrystalline powder of Fe—Al, Ni—Al intermetallic compounds [10–12], M50 steel [13] and Fe based metglas [14]. Later, a wave of research, focusing on the preparation of nanostructured powder of pure Al, Al alloy and Al-MMCs, was emerged [8,15–19]. For pure Al and Al alloys, the existing powder consolidation technologies include some conventional powder metallurgy techniques, such as HIP [19,20], hot rolling [21], spark plasma sintering [22], vacuum hot pressing, etc. [23,24]. However, these consolidation technologies often affect the final structure of the powder due to high processing temperature.

Cold spraying (CS) is an emerging solid-state additive manufacturing technology, which mainly depends on the powerful kinetic energy of the impacting particles to achieve the coherence between the powder particles. The low-temperature solid-state processing makes CS an attractive technique to spray thermally sensitive materials, such as nanostructured, amorphous materials etc. [25]. Nearly a decade ago, Ajdelsztajn et al. first used cryomilled feedstock powder to prepare nanostructured coating through CS [26,27]. Later on, many researchers carried out the follow-up studies on pure Al [28], Aluminum alloy

* Corresponding authors.
E-mail addresses: jqwang11s@imr.ac.cn, (J. Wang), tyxiong@imr.ac.cn (T. Xiong).

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2009 [29], Aluminum alloy 2618 [30], Aluminum alloy 5083 [26,31,32], Al-7.6 at.% Mg [33], Aluminum alloy 6061 [31], Aluminum alloy 7075 [34], B_4C-Al12Si MMCs [35], CNT-Al MMCs [36] etc. These studies demonstrate successful fabrication of the nanostructured coatings that retain the inherent structural features of the feedstock powder. The coatings exhibited ultra-high hardness, along with increased porosity compared with the coatings fabricated by gas atomization powder.

In CS, kinetic energy is transferred to powder particles from the supersonic gas stream coming from the converging-diverging type gun nozzle. Therefore, the kinetic energy storage capacity of the powder is of the paramount importance which influences the quality of the deposit to a great extent. This capacity depends upon the density, size distribution and morphology of the starting powder. Meanwhile, an optimal size distribution range is required for different kind of powders to achieve high deposition efficiency during CS. Many factors influence the optimal size distribution for a powder. These include; density of the particles, their deformability, and the thickness of surface oxide film of the starting powder. Therefore, it is imperative to carefully tailor the morphology and size distribution of the feedstock powder to suit CS process. Unfortunately, traditional cryomilling operation has relatively low control over powder's geometric characteristic, particularly for the powders having FCC crystal structure. This shortcoming makes this process unattractive to produce UFG/nanostructured feedstock for cold spraying process.

In past, no serious attention was paid to the morphology of cryomilled feed stock powder. Consequently, benefits associated with the cold spraying process could not be achieved completely. For instance, the morphology of the starting pure Al powder is completely changed from spherical to lamellar structure as a result of traditional cryomilling process. This kind of powder morphology is not suitable for CS process to achieve high deposition efficiency and good tamping effect. Therefore, the present study was carried out with the aim to explore the influence of cryomilling process on geometrical characteristics and grain refinement of pure Al powder at various milling conditions. The geometrical characteristics of Al powder was controlled by using various combinations of the milling media (i.e. steel balls) of different sizes.

2. Material and methods

Commercially available atomized pure Al powder with particle size <20 μm (~600 mesh) was milled in a modified pot vibration mill with stainless steel balls. Nanocrystalline Al powder was successfully produced by cryomilling at 100 K. The vibration frequency and the amplitude for the milling process were kept at 45 Hz and 5 mm, respectively. Seven different sizes of stainless steel balls and as-atomized stainless steel powders were used as the milling media. The balls were designated as D_{10}, D_7, D_5, D_1, $D_{0.5}$ $D_{0.3}$ $D_{0.1}$, whereas the subscript shows the diameter of the ball in millimeter. The other parameters of the milling process are summarized in Table 1.

The size distribution of various powders was made using Malvern mastersizer 2000. The morphology of the powders was observed by FEI Inspect F scanning electron microscopy (SEM). Transmission electron microscopy (TEM) was performed using 200 kV FEI Tecnai F20 equipment. Microhardness measurements were performed on the cross-section of different powders using a Vickers microhardness tester (AMH43, Leco, USA) under a 10 g load and 15 s holding time. For each powder sample, at least 7 indentations were taken at random locations and their average values as well as the standard deviation were calculated.

3. Results

The SEM micrograph of the as-received Al powder is shown in Fig. 1. The as-atomized powder shows uniform equiaxed spheres with smooth surface morphology.

3.1. Properties of the cryomilled powders

The SEM micrographs of Al powder cryomilled at different milling conditions (in the presence of PCA) are shown in Fig. 2. It can be observed that by adding PCA in the vibration mill, the cold welding of the particles is completely inhibited. Under cryomilling conditions, powder particles experienced severe deformation due to the repeated

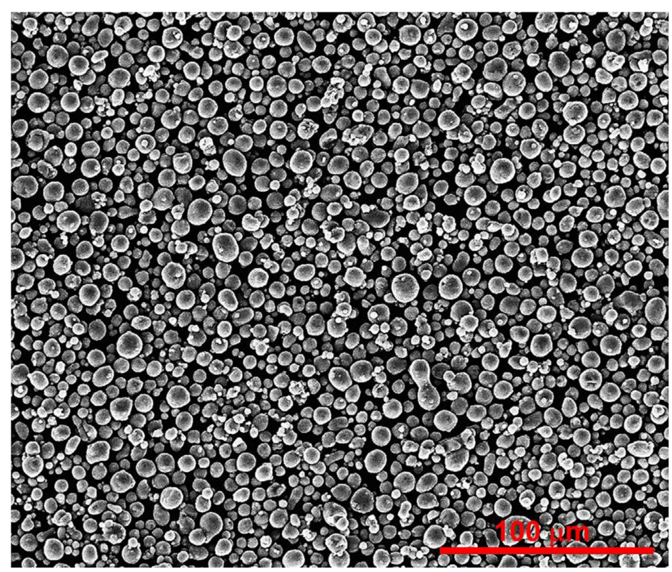

Fig. 1. SEM image of as-received Al powder.

Table 1
Summary of different parameters for modified vibratory milling process.

Powder designation	PCA (Stearic acid, wt%)	Milling time (hr)	Milling balls		Ball-to-powder weight ratio	Filling ratio (%)
			Combination	Dissimilar balls weight ratio		
1–1	5	2				
1–2	5	4				
1–3	5	7				
1–4	5	13	D_{10}, D_7, D_5	1:1:1	15:1	60
1–5	1	4				
1–6	0.5	4				
1–7	0.1	4				
2–1		2	D_{10}, D_7, D_5	1:1:1		
2–2		2	D_{10}, D_7, D_5, D_1	1:1:1:1		
2–3	–	2	D_{10}, D_7, D_5, $D_{0.5}$	1:1:1:1		
2–4		2	D_{10}, D_7, D_5, $D_{0.3}$	1:1:1:1		
2–5		2	D_{10}, D_7, D_5, $D_{0.1}$	1:1:1:1		