Powder Technology 284 (2015) 149–158

Contents lists available at ScienceDirect

Powder Technology

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

Effect of ball size on steady state of aluminum powder and efficiency of impacts during milling

S.S. Razavi-Tousi *, J.A. Szpunar

Department of Mechanical Engineering, University of Saskatchewan, S7N 5A9 Saskatoon, Saskatchewan, Canada

ARTICLE INFO

Article history:
Received 21 January 2015
Received in revised form 12 April 2015
Accepted 13 June 2015
Available online 20 June 2015

Keywords:
Mechanical alloying
Microstructure
Milling parameters
Impact energy
Charge ratio

ABSTRACT

The concept of steady state milling time was examined for ball milling of aluminum powder. Four different set-ups of balls were used while the mill speed and charge ratio were kept fixed. Different criteria (morphology of particles, average particle size, deviation from the average particle size, lattice imperfections and change in crystallographic orientation) were used to study structural evolution of the milled particles and to compare the steady state time for the different milling conditions. Results showed that different criteria may not determine the same steady state time, however, all criteria were consistent in comparing efficiency of the different milling conditions. Moreover, it was found that at a given mill speed and ball to powder ratio (i.e. at a given consumed energy), a change in balls size and filling ratio of vials can improve milling efficiency. Finally, the effect of energy of each impact and the collective energy of all impacts were discussed.

1. Introduction

During past 20 years, high energy ball milling has extensively been utilized as a versatile method to produce variety of advanced compound powders. The main difference between high energy milling by planetary ball mill, Spex, Attritor mill, etc. and conventional milling is that the former applies much larger doses of energy to particles over time [1]. This large input energy makes high energy mills capable of producing materials with special properties that could not be synthesized by other methods, mechanochemistry is a typical example for this capability [2]. On the other hand, the large amount of energy consumed by high energy mills is potentially a burden in industrial application of this method. That is because the electrical energy spent for production of the powders by high energy mills is added to the final price of the products.

Researchers have adopted two ways to decrease the energy needed for production of powder products by high energy mills. The first is to find the minimum time needed to produce the desired products. For the products that are synthesized by a reaction, whether the reaction occurs gradually or suddenly during milling, the minimum milling time can easily be determined by the time that is required for whole amount of reactants to be transformed/reacted to form the desired products [3–6]. However, for production of nano-crystalline powders by severe plastic deformation where no reaction is involved, or for activating a powder product for a reaction, it is not straightforward to determine the needed minimum milling time. Accordingly, researchers introduced the "steady state time", which is defined as the milling time after which further milling has negligible effects on the powder products [1,7–12]. Changes in size and shape of particles, preferred orientation and presence of lattice imperfections (e.g. grain boundaries, dislocations etc.) can be used as the criteria for determination of the steady state.

The second way of decreasing the energy consumed by high energy mills is to make the milling more efficient in a way that the system reaches the steady state in a shorter period of time. Changes in ratio of rotation of vials compared to disk [13,14], ball size [4,15–18], mill speed [13–15,17,19–23], filling ratio of vials [4,15,18–21,24] and charge ratio [4,6,25–27] have been studied to optimize the milling process.

Using a particle swarm optimization method, Abdellahi et al. found that optimizing milling parameters has a significant effect on the milling products [28]. Sato et al. conducted a discrete element method simulation and compared it with their experimental results and found that a filling ratio of about 50% is the optimum for planetary ball mill [15]. Ashrafizadeh et al. drew a similar conclusion regarding the filling ratio using the simulation results [19]. Ward et al. found that an increase in ball size may increase or decrease the efficiency of milling, depending on other milling parameters [4]. High speed video recording by Rogachev et al. suggested that different ball motion patterns in vials can be obtained by changing the rotational speed of vial [29]. Mio et al. and Rosenkranz et al. found that the pattern of ball movement in vials is an effective parameter in the efficiency of milling [13,24]. Lv et al. found that the particle size of the produced powder by ball milling changes by charge ratio [30]. Delogu et al. considered the milling time needed for occurrence of mechanochemical reactions as the criterion to evaluate the milling efficiency [6,31,32]. They developed a model

* Corresponding author. Tel.: +1 306 290 9084.
E-mail addresses: ser105@mail.usask.ca, s.razavitousi@gmail.com (S.S. Razavi-Tousi).

http://dx.doi.org/10.1016/j.powtec.2015.06.035
0032-5910/

and correlated the kinetics of milling to the charge ratio or number of balls. Their results showed the time needed for occurrence of a mechanochemical reaction is correlated to C_R^{-2}, where C_R is the charge ratio [6]. On the other hand, Balaz et al. found that ignition time of a mechanically-induced self-sustaining reaction is correlated to C_R^{-1} [33].

Although there is no agreement in literature on the effect of some of the mentioned variables, the unanimous conclusion is that a higher speed and a larger charge ratio result in a faster formation of final products. However, a higher speed of a mill requires higher energy inputs. A larger charge ratio also means that for a given amount of feed (balls + powder), a less amount of powder is produced. Therefore, although an increase in either mill speed or charge ratio decreases the steady state time, it still may increase the price of final products.

In this work, we aimed to improve the efficiency of high energy ball milling without manipulating either mill speed or charge ratio. In this way, we attempted to shorten the steady state time for a given energy input. We examined steady state using different criteria applied to different milling conditions. The most efficient milling condition will be established and the justification of the proposed method will be discussed.

2. Experimental procedure

2.1. Powder preparation

Aluminum powder of 99.8% purity (MFCD00134029) was provided by Alfa Aesar Company. Stearic acid (97% purity, Fisher Scientific-AC17449-0010) was used as the process control agent (PCA) and 3 wt% of it was mixed with the aluminum powder prior to milling. High energy ball milling was done in a planetary ball mill (Torrey Hills-ND2L) with stainless steel cups (285 ml capacity) and balls in an argon atmosphere. Charge ratio was 30:1 and mill speed was maintained at 200 RPM for durations of 0.25, 0.5, 1, 2, 4, 7, 11 and 19 h. Table 1 shows the four different mixtures of balls that were used in this study. We name them vial. 1, vial. 2, vial. 3 and vial. 4. One can see that for vial. 1–4 the mill speed and charge ratio are constant, while the number and size of the used balls vary.

2.2. Scanning electron microscopy

A Hitachi SU6600 field emission scanning electron microscope (SEM) examined the powders after ball milling. Particle size of the powders was calculated by software written in Visual Basic using several SEM images. The surface weighted average Eq. (1) was calculated and reported as the average particle size.

$$D_s = \frac{\sum_i n_i D_i^3}{n_i D_i^2} \tag{1}$$

where D_i is diameter of the particle i, n_i is number of particles with size D_i and D_s is surface weighted average.

Table 1
Milling condition used for vial. 1–4.

Vial no.	6 mm (#)	10 mm (#)	16 mm (#)	18 mm (#)	Total balls weight (gr)	Al powder (gr)	Mill speed (rpm)	Charge ratio
1	202	19	28	8	875	28	200	30
2	–	–	34	–	585	20	200	30
3	337	–	–	–	300	10	200	30
4	225	21	–	2	318	10.6	200	30

Standard deviation from the average particle size was used as a measure of particle size distribution:

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(D_i - D_s)^2} \tag{2}$$

where N is the total number of particles and σ is the standard deviation from the average particle size.

To examine the cross section of the particles, we added a small amount of the powder to a conductive carbon resin powder, mixed and mounted it. We grinded the mounted samples by abrasive grinding papers from 600 to 1600 grit followed by additional polishing by diamond pastes. Finally to get high quality surfaces, the samples were polished using a Hitachi IM4000 Ar ion milling system. A Nordlys Nano Oxford detector collected electron backscattered diffraction (EBSD) patterns at a voltage of 20 kV with a tilt degree of 70°.

2.3. X-ray analyses of powders

Microstructure of the powders was characterized by a Bruker D8 Discover X ray diffractometer with a chromium target. In order to obtain improved peak parameters, X-ray diffraction (XRD) peaks were analyzed as follows: $K\alpha_2$ stripping was done applying the Rachinger method [34], considering $K\alpha_1 = 2.289760$ and $K\alpha_2 = 2.293663$. It was assumed that the $K\alpha_1$ and $K\alpha_2$ line profile are identical in shape and not necessarily symmetrical, and that the α_2 peak is half of the intensity of the α_1 peak, and is shifted from it toward larger angles by:

$$\Delta 2\theta = 2\tan\theta\left(\frac{\Delta\lambda}{\lambda}\right) \tag{3}$$

where Δλ is the dispersion separation $\lambda(\alpha_2)$–$\lambda(\alpha_1)$ in angstroms.

Pseudo-Voigt profile fitting was used to decompose a complicated powder diffraction pattern, assuming that it can be represented by the weighted mean between a Lorentz and a Gauss function:

$$G_{ik} = \gamma\frac{C_o^{0.5}}{H_k\pi}\left[1 + C_o X_{ik}^2\right] + (1-\gamma)\frac{C_1^{0.5}}{H_k\pi^{0.5}}\exp\left[-C_1 X_{ik}^2\right] \tag{4}$$

where $C_0 = 4$, $C_1 = 4 \times \ln 2$, H_k is the full-width at half-maximum (FWHM), $X_{ik} = (2\theta_i - 2\theta_k) / H_k$. γ is a refinable "mixing" parameter describing the amount of Gaussian profile versus the amount of Lorentzian profile; and thus describing the overall profile shape.

3. Results and discussion

3.1. Morphological changes

Figs. 1–4 show the effect of milling time on the morphology of aluminum particles in vial. 1–4, respectively. The trend of the changes in morphology is almost the same for all the vials; the particles were initially flattened and cold welded, followed by fragmentation and formation of equiaxed particles. Impacts at the beginning of milling deform the particles and morphology of the particles changes to disk-shape. Besides, cold welding occurs and particles grow in size. Further milling and deformation increase hardness of the particles, therefore, formability of particles decreases and particles tend to be fractured by impacts rather than being deformed or cold welded. The final shape and size of the particles (steady state) are determined by equilibrium between cold welding and fracture mechanism.

Considering Figs. 1–4, one can realize that the change in the milling condition (Table 1) does not change the overall trend of evolution of particles toward a steady state, but it changes the time by which a system reaches steady state. For example in the case of vial. 1 & 2, no flake-