



Contents lists available at ScienceDirect

Advanced Powder Technology

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

Original Research Paper

Optimum milling parameters for production of highly uniform metal-matrix nanocomposites with improved mechanical properties

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ARTICLE INFO

Article history:

Received 9 January 2018
 Received in revised form 14 June 2018
 Accepted 7 July 2018
 Available online xxx

Keywords:

Optimization
 Electron microscopy
 Hardness
 Composites
 Powder methods

ABSTRACT

In the present paper, a system dynamic model is presented to predict the final particle size of milled powder during ball milling process. The presented model is used to obtain the optimum ball size, milling speed and milling time that achieve the best particle size reduction of metal-matrix nanocomposites. Parametric study is performed using the presented analytical model to study the influence of ball size and milling speed on the milling efficiency. The predictions of the presented model are validated with experimental results done during this work for Cu-5%ZrO₂ nanocomposite and others available in the literature. The results show that the milling time required to achieve the steady state condition for Cu-5% ZrO₂ nanocomposite is 15 h. At 15 h of milling, ZrO₂ particles are highly uniform distributed in Cu matrix and the microhardness is increased from 75.4 HV for Cu to 197.6 HV for Cu-5%ZrO₂ nanocomposite. After 15 h, the particle size reduction rate is too low and the hardness improvement rate is too low as well (204.1 HV after 20 h milling) which make the milling process after 15 h is not appreciable.

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1. Introduction

Currently, the demands for nanocomposite materials due to their superior mechanical properties are increasing when compared to monolithic materials/conventional coarse-grained metal matrix composites [1,2]. However, the method of fabrication and reinforcement volume fraction are attracting researchers' attention in this field. Copper is one of the most promising metals for such an application. However, its strength must be increased in order to meet the design requirements for high-temperature applications. Conventional strengthening mechanisms such as cold working, precipitation and dispersion hardened alloys have been used for this purpose [3–5]. However, the former two methods were not able to satisfy the high temperature strength demands, because of the effects of recrystallization and precipitate coarsening or dissolution. An alternative promising technique to get the desired properties is dispersion strengthening of zirconia (ZrO₂) in copper. Since ZrO₂ possesses high mechanical properties, the combination of mechanical and electrical properties of ZrO₂ and Cu leads to application of dispersive strengthened Cu as welding electrodes, lead frames, accelerator electrodes and electrical connectors [6–8].

Mechanical alloying (MA) is a widely used technique in synthesizing nanocrystalline materials and also it has been used to prevent the reinforcement clusters or agglomerates on the matrix, especially in the case of small size reinforcement particles that produce uniform dispersion in the matrix [1,4]. The homogeneous dispersion of fine reinforced particles in a fine grained matrix is beneficial to the mechanical properties of metal matrix composites. The process of MA is based on imparting a severe plastic deformation (SPD) on the powder using high-energy ball milling technique [9–11]. Using this method, an extremely high strain is imparted on the material and the structural refinement occurs by shear and fracture of phase mixtures. This kind of SPD leads to formation of a nanograin structure. The MA process consists of repeated cold welding, fracturing and cold welding of powder particle mixture in a high-energy ball mill which lead to a reduction in the powder particle size [1,2,12].

Fig. 1 shows a schematic drawing of a planetary ball milling machine with 4 vials. In planetary ball milling machine, the disk and the vials rotate in opposite directions to make a turbulent motion of the balls inside the vial. The particle size reduction was mainly resulted from the complicated dynamic interaction of the balls with turbulent powders during the ball mill process, in which powder particles are entrapped between collided balls [13,14]. Many collision scenarios can occur during the milling process lead to particle size reduction. Collision between two balls

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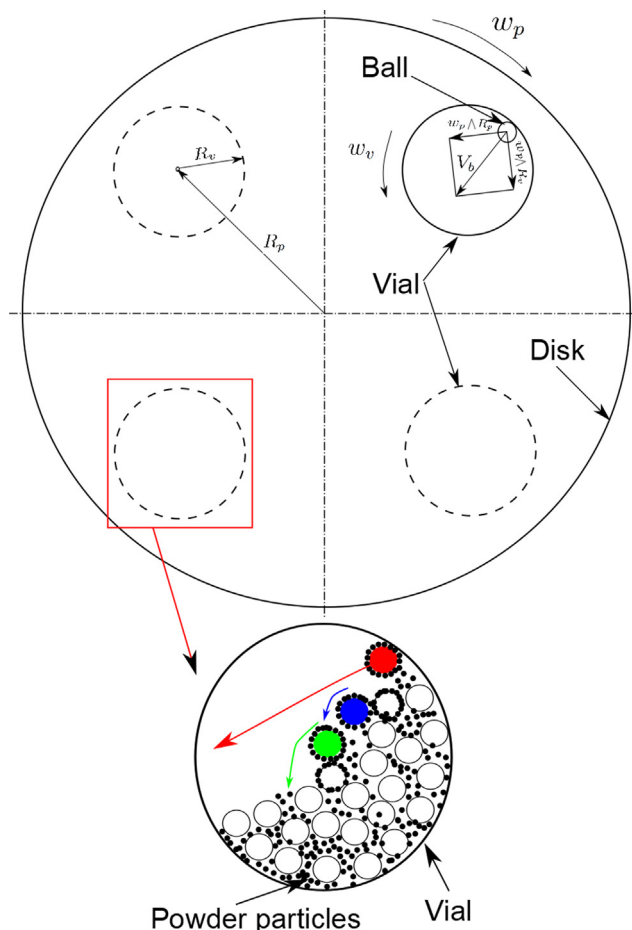


Fig. 1. Scheme of a planetary ball milling machine containing 4 vials showing the possible collision mechanisms during milling process. Adapted from [15] and modified in the present study.

moving inside the vial (blue¹ ball in Fig. 1), between one movable ball and other immovable one in the powder medium (green ball in Fig. 1), between a ball and the vial wall (red ball in Fig. 1) and between two powder particles [15]. It is believed that the collisions between a movable ball and other immovable balls in the powder medium are the most effective collision in the particle size reduction [13]. Also, the collision between the movable balls and the vial wall is more effective than the collision between movable balls and each other and also more efficient than the collision between the powder particles [14]. Hence, the main source of the particle size reduction is the collision energy between the collided balls. This collision energy basically depends on the ball size and mass and its velocity [15].

Therefore, the main objective of the present work is to obtain the optimum milling parameters which achieve the highest rate of particle size reduction. The analytical model used for the optimization process is implemented and validated by comparing its prediction with experimental results available in the literature and other results obtained during this work. The effects of milling speed and ball size have been analyzed to highlight the influence of both parameters on the milling efficiency. Finally, the optimum milling time, ball size and milling speed is determined by the applying the presented analytical model. Cu-5%ZrO₂ nanocomposite was manufactured using MA technique with the obtained optimum parameters and mechanically characterized to check the validity of the model.

2. Materials and experimental procedure

Commercial pure copper powder (Cu) with an average particle size of 80 μm and 99.5% purity and Zirconia (ZrO₂) (99.9% purity and 5 μm average particle size) supplied by Sigma Aldrich, Egypt, were used as raw material for composite fabrication. The composite powders of Cu with 0 and 5 wt% ZrO₂ were milled in a Fritsch planetary ball mill using a hardened stainless steel vial and hardened steel balls of 10 mm in diameter. A 500 ml vial was used to prepare the composite where the total amount of powder was 25 g for each experiment. The ball to powder mass ratio was constant and equal to 20:1 and the vial rotational speed was 300 rpm. These parameters, ball size and rotational speed were determined based on the presented optimization algorithm in Section 5.1. Stearic acid (3 wt%) was used as the process control agent to prevent excessive cold welding of powder particles. Four different milling times were considered for this study, 5, 10, 15 and 20 h, with 15 min interruption after each 2.5 h milling to avoid the temperature increase. The produced powders after milling were investigated by using Scan Electron Microscope (SEM) Model Quanta 250 FEG (Field Emission Gun) attached with EDX unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 kV. The average particle size of the produced powders was calculated by image processing software, Image J, using several SEM images. At least 3 SEM images was analysed for each batch of the powder, in each SEM image 10 particles were measured and the standard deviation of the obtained measurements was calculated. In all cases, to have a good representation of the average particle size of the milled powder, the size of 40–50 particles was measured and the average particle size is considered. Some of the prepared samples were inspected using JEOL 3010 high-resolution transmission electron microscope (HR-TEM).

X-ray diffraction (XRD) inspections were carried in a Rigaku-DXR 3000 X-ray diffractometer using Cu K α radiation ($k = 0.15406 \text{ nm}$) at 30 kV and 30 mA settings. The XRD patterns were recorded in the 2θ range of 20–80° with a step size of 0.02° and a scanning rate of 1.5 degs/min. The crystallite size of milled powders was estimated by XRD peak broadening using William-Hall method as follows: $D = \frac{0.9\lambda}{B \cos \theta}$, being D , B , λ and θ are crystallite size, full width at half maximum (FWHM), the wave length and peak position, respectively.

After the powder characterization, the prepared powders were cold compacted at 500 MPa in cylindrical die with 10 mm diameter and then sintered for 2 h at temperature 900 °C. The consolidated samples were then polished up with different grades of silica paper and diamond paste. The distribution of the reinforcement particles in the matrix was examined using EDX technique.

Hardness of the prepared samples, Cu and Cu-ZrO₂, was measured with micro-hardness Vicker indenter by applying a load of 10 g for 15 s over the specimen in-plane section, ensuring that the load is sufficient to obtain indents much smaller than the powder particles. To obtain the accurate value of the powder hardness, 10 measurements were performed for each sample and with Grubbs test, outliers were discarded from the population.

3. System dynamic model

The ball milling process is governed by a large number of parameters which influence the efficiency of the milling process [16]. Some of these parameters have a direct effect on the output of the experiment while others have an indirect effect which affect each other. It is not easy to directly provide analytical models to predict the particle size after the milling process taking into account all the parameters affecting the experiment. Hence, the

¹ For interpretation of color in Fig. 1, the reader is referred to the web version of this article.

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