Powder Technology 274 (2015) 44–52

Contents lists available at ScienceDirect

Powder Technology

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

Experimental investigation of milling regimes in planetary ball mill and their influence on structure and reactivity of gasless powder exothermic mixtures

A.S. Rogachev [a,b,*], D.O. Moskovskikh [a], A.A. Nepapushev [a], T.A. Sviridova [a], S.G. Vadchenko [b], S.A. Rogachev [b], A.S. Mukasyan [c]

[a] National University of Science and Technology, "MISIS", Moscow 119049, Russia
[b] Institute of Structural Macrokinetics and Materials Science, RAS, Chernogolovka 142432, Russia
[c] Department of Chemical and Biomolecular Engineering, University of Notre Dame, Notre Dame, IN 46556, USA

ARTICLE INFO

Article history:
Received 14 October 2014
Received in revised form 30 December 2014
Accepted 2 January 2015
Available online 9 January 2015

Keywords:
High energy ball milling
Mechanical activation
Planetary ball mill
Ball motion
High energy density materials
SHS

ABSTRACT

The dynamics of ball motion during high energy planetary ball milling was investigated in-situ using high speed video recording. The trajectories and velocities of the balls were determined for different milling conditions. Correlations of these parameters, with both microstructure and reactivity of various as-milled powder mixtures (Ni–Al, Ti–Si, Si–C), were revealed. It was shown that all of the main theoretically predicted milling regimes – cascading, cataracting and centrifugal – can be achieved by varying the rotation speed of the vial. The conclusion is made that shear deformation plays a major role in the transformation of the microstructure and reactivity of these powder mixtures. According to this observation, the cascading regime appears to be the most effective for controlling the properties of the mixtures.

1. Introduction

High-energy ball mill (HEBM) is widely used for grinding, alloying and mechano-chemical synthesis of various materials, including nanomaterials [1–3]. It was also found that HEBM of reactive systems (i.e. mixtures of two or more powders that can react in a self-sustained mode) results in a decrease of the reaction onset temperature and an increase of the reaction rate [4–7]. Thus, it is concluded that such a mechanical treatment "activates" the reactive systems. During the last 15 years, special attention was paid to, the so-called, mechanically activated self-propagating high-temperature synthesis (MA-SHS) [8–12]. SHS reactions allow for the fabrication of different materials by means of the "solid flame" (gasless) type of processes in a heterogeneous mixture of powder reactants (cf., [13,14]). An essential requirement for such reactions is that they must generate enough heat in order to proceed in the self-sustained mode. The combination of HEBM and SHS significantly expands the capabilities of both methods, because such a mechanical treatment decreases the critical threshold for heat release. Weakly exothermic mixtures (e.g., Si + C), for which the self-sustained reaction could not be accomplished under conventional conditions, become combustible after HEBM [15]. The mechanism of this effect is still not well understood. Our recent investigation suggests that structural modification of the reactive media, including both nano-scale mixing of the reactants and sometimes the formation of nuclei of new phases during HEBM, may be responsible for the enhancement of reactivity in the Ni + Al and Ti + C systems [16,17]. Severe deformation of metals and the formation of sub-micrometer, or nano-scale, composite particles during ball milling have been observed in many works [9,10,12]. In order to understand how these HEBM-induced structures formed, it is critical to know the parameters of the ball movement in the grinding vessel.

The trajectories of the balls inside the vessel during HEBM were studied in numerous theoretical and few experimental works. According to theoretical analyses, depending on the rotation speed, friction coefficient, ball diameter, and other parameters: the balls can roll and slide along the inner surface of the grinding chamber, move along free-fall trajectories, make periodic circular, or chaotic swirling motions (cf. [18–20]). A variety of parametric maps were modeled to show the correlations between the trajectories of the balls and planetary mill rotation regimes (cf. [21]). Unfortunately, the validation of these theories is hardly possible due to the lack of direct experimental data on the real ball behavior during milling. Moreover, the available experimental observations often contradict the theoretical predictions. For example, most of the models consider impacts between the balls as the main

* Corresponding author at: Institute of Structural Macrokinetics and Materials Science, RAS, Chernogolovka 142432, Russia.
E-mail address: rogachev@ism.ac.ru (A.S. Rogachev).

http://dx.doi.org/10.1016/j.powtec.2015.01.009
0032-5910/

factor, which influences the powder structure. However, the first direct photo- and high speed video-recording of the ball motion led to the conclusion that the HEBM process should be primarily described in terms of attrition and wear and not in terms of impact [22]. For example, the direct high-speed video recording of the ball motion made by the camera installed on the rotating sun wheel above the jar, showed only rolling and sliding of the balls for all investigated parameters of the considered planetary mill, while the free falling regime has not been observed [23].

In this work we study the ball motion in-situ in the planetary mill, depending on the parameter $K = w/W$, where w and W are the rotation speeds of the grinding vessel and the sun wheel, respectively. High speed video recording, followed by frame by frame computer treatment of the obtained movies, allows us to retrieve the trajectories of the balls and measure both their instantaneous velocities and accelerations for different milling conditions. The same HEBM regimes were applied to three separate binary reactive powder mixtures, i.e. Ni–Al (ductile–ductile), Ti–Si (ductile–brittle) and Si–graphite (brittle–brittle). The morphology and reactivity of these systems were investigated as a function of parameter K. The correlations between the planetary ball mill regimes and the properties of the as-milled reactive powder mixtures were identified and discussed.

Thus, the goal of this work is to obtain direct experimental data on the correlations between the different high energy planetary ball milling regimes of the reactive powder mixtures, the morphology of as-milled particles, and the reactive properties of these mixtures.

2. Experimental procedure

A laboratory-scale planetary ball mill "Activator-2S" (Novosibirsk, Russia) was used in this work. Two vertical grinding steel vessels (vials), with inner volumes of 250 ml apiece, are placed on the sun wheel, as shown in Fig. 1. Two independent electric motors drive the sun wheel and the vials with the rotating rates W and w, correspondingly. A computer controls the rotation speed through two frequency converters. The operational parameters used in this research are listed in Table 1. Note that for this mill, the rotating directions of the sun wheel and the grinding vials are opposite, therefore, the parameter $K = w/W$ has a negative value. However, for the sake of convenience, we use an absolute value of the rotating velocities for determination of K value. As can be seen from Table 1 we kept the velocity of the sun wheel constant ($W = 694$ rpm) and changed K by varying the velocity of the vial (w) from 0 to 1388 rpm. All other parameters remained constant during all experiments. The values of the pre-set rotating speeds were verified with the high-speed video camera.

In the video recording experiments, the steel lids of the vials were replaced with transparent acrylic glass lids, and four LED lamps were used for illumination of the sun wheel and the vials. The high speed video camera Phantom Miro M310 (Vision Research Inc., USA) was placed above the mill, and the lens of the camera was directed along the vertical axis of the sun wheel. The video frame at the Fig. 1a represents the view field of the camera. A recording rate of 2000 frames per second was used in all experiments. In order to monitor the trajectories of the balls inside the grinding vessels, computer treatment of the video records was made. Two square windows were selected, as shown in Fig. 1b. These windows rotated around the sun wheel axis with the same rate as the grinding vials. Thus, axis of each vial remained stationary relative to the window, i.e. as if the video camera was physically installed at the sun wheel on the top of the vial. This procedure was accomplished by the use of a specially developed computer program,

Table 1
Parameters of the HEBM used in this work.

Radius of the sun wheel, R	0.104 m
Radius of the grinding chamber, r	0.040 m
Distance between the axle of the sun wheel and the axle of the grinding chamber, R-r	0.064 м
Parameter M = (R-r)/r	1.6
Radius of the milling ball, r_b	0.003 m
Mass of the milling ball (steel), m_b	0.8 g
Rotating speed of the sun wheel, W	694 min^{-1}
Rotating speed of the grinding chamber, w	0–1388 min^{-1}
Parameter K = w/W	0–2.0
Volume fraction of the balls inside the grinding chamber	0.35
Duration of milling	3–15 min

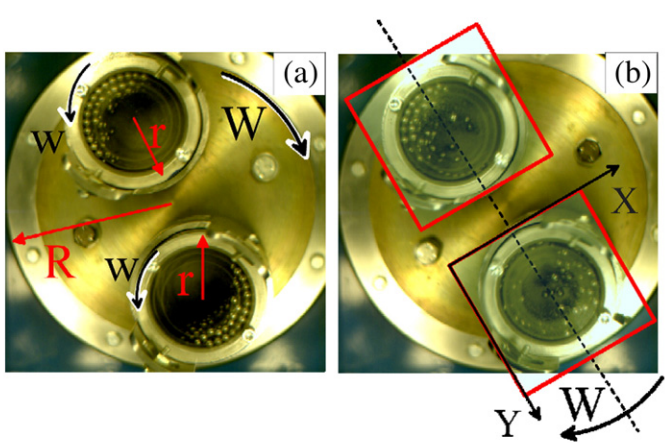

Fig. 1. High-speed video frames of the rotating planetary mill. K = 1.0 (a) and K = 1.9 (b).

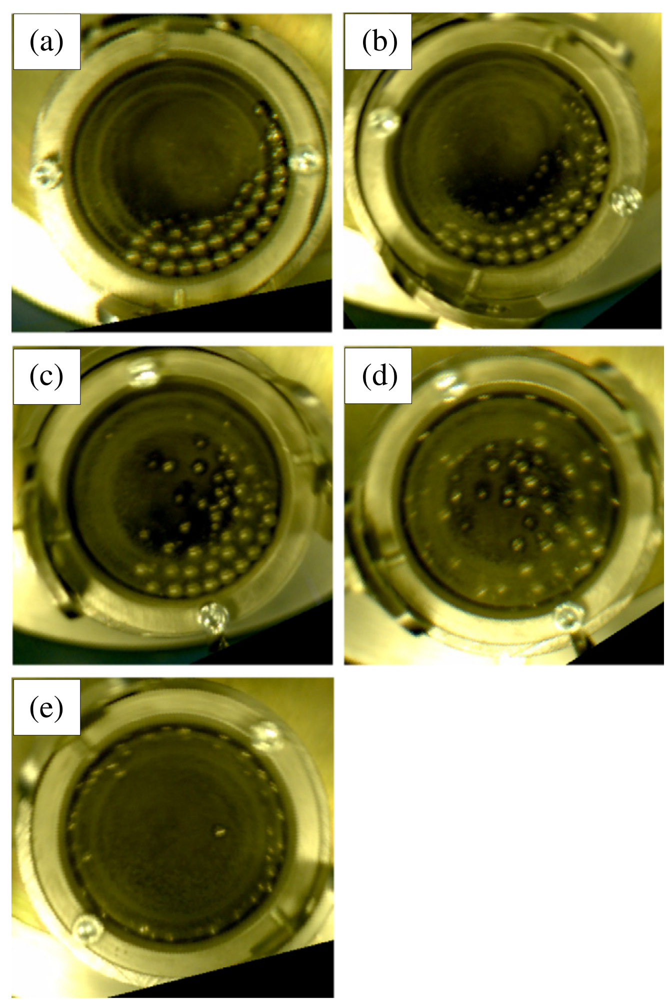

Fig. 2. Instant ball distribution in the grinding vials depending on K values: K = 1.0 (a); 1.5 (b); 1.8 (c); 1.9 (d); 2.0 (e).