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Effect of jar shape on high-energy planetary ball milling efficiency: Simulations and experiments

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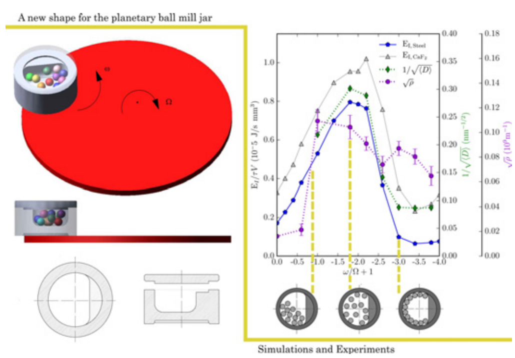
HIGHLIGHTS

- An innovative jar shape for the planetary ball mill is proposed and characterized through simulations and experiments.
- Increased high velocity collisions along impact axis enhance comminution with respect to the conventional jar.
- The range of velocity ratios granting the maximum grinding efficiency is wider with respect to the conventional jar.
- Information from X-Ray diffraction analysis of a milled test material support and validate simulation results.

GRAPHICAL ABSTRACT



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ABSTRACT

Enhanced comminution in a planetary ball mill was achieved by suitably re-designing the jar shape. Compared with a traditional cylindrical vial of circular cross-section, the new jar was modified internally to have a flat wall portion resulting in a half moon cross-section. Results from simulations using a multibody dynamics software, suggest that this geometry increases the number of high-velocity collisions with energy exchange along the axial direction, deemed as more effective in the comminution process. X-ray diffraction line profiles of calcium fluoride (CaF_2) ground in the two jars under equivalent conditions were used to obtain information on the microstructure resulting from the milling process and validate the modelling results. A better homogeneity and a faster reduction of crystallite size were achieved using the new design compared to that using the standard cylindrical vial design. Optimal operating conditions, in terms of jar-to-plate angular velocity ratio, are correlated and discussed according to the model predictions.

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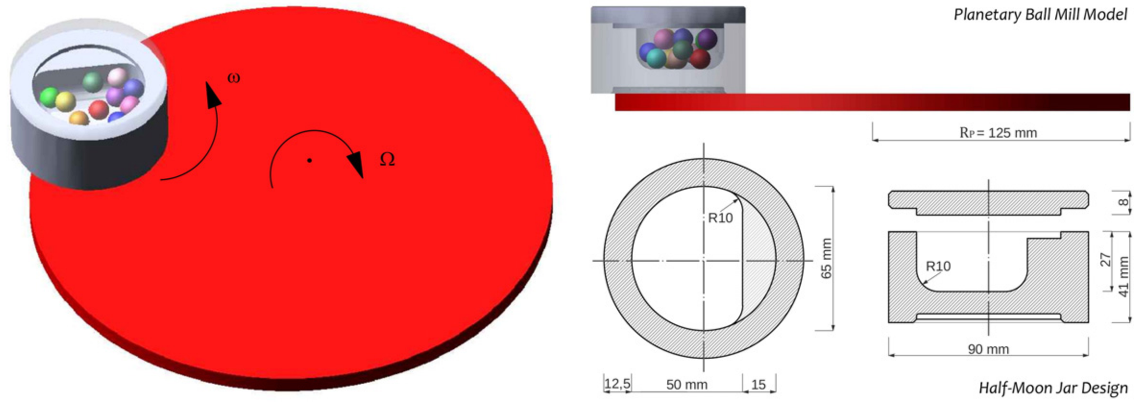

Fig. 1. Planetary ball mill installing the half moon jar and definition of rotation (ω) and revolution (Ω) angular velocities. The design of the new half-moon shaped jar is also reported.

1. Introduction

Planetary ball milling is a widespread and versatile technique for the structural and microstructural properties tuning of almost any kind of material. It is often used to grind materials down to the nanoscale, generally providing both comminution and incorporation of defects by extensive plastic deformation, thus supplying the conditions for mechanical activation [1,2]. Remarkable examples are the mechanical alloying of metals as well as the direct and indirect synthesis of ceramics [3–5]. The method has also proved to be effective in the mechanical exfoliation of bulk systems, for the large-scale production of two-dimensional nanostructured materials - such as graphene and boron nitride nanosheets [6,7], nowadays of utmost scientific and technological interest. Although rather simple in terms of geometry and working principles (see Fig. 1 and *e.g.* [8]), the versatility and efficiency of the planetary ball mill depend very much on the appropriate tuning of a multitude of milling variables. Among the others, the number and size of the balls, the jar geometry and the velocity of revolving parts should certainly be mentioned. The setup parameters strongly influence the ball trajectories, which, in turn, determine the nature and magnitude of the impulsive forces transferred by milling media collisions. These forces can be divided into normal and shear components: the former should promote fracture while shear components induce plastic deformation [9,10,6].

As an alternative to extensive experimental testing, deemed as expensive and time-consuming to the point of being most often unfeasible, a dynamic-mechanical model of planetary ball milling was adopted [11] to fine-tune milling parameters and establish correlations with the end-product. This model, based on a simple contact scheme depending on a few easily estimable parameters, supplies quick and accurate predictions of system efficiency under different operating conditions and is, in principle, valid for any material of interest. The effect of jar (ω) and plate (Ω) velocities was investigated, providing a detailed picture of the kinematics and dynamics of the milling bodies, together with a description of contact events, supporting an outright understanding of ball motion regimes. An assessment of the kinetic energy available, both along normal and tangential directions, was obtained and results were validated against experimental data. In particular, best milling conditions, corresponding to the highest impact energy, were found to correlate with the most disordered ball motion, which develops within a well-defined range of jar-to-plate velocity ratios ($\omega/\Omega+1$).

Beyond the influence of plate and jar angular velocities, the present work investigates the effect of jar shape on the process efficiency. Particularly, the ratio of normal-to-shear transferred loads was considered to be an essential parameter in the design of end product characteristics, in terms of structure and microstructure. To increase the number of collisions with high-normal-velocity component, a new type of jar was designed. Simulation results, obtained using the above-mentioned multibody dynamics model, were analyzed in terms of kinetic energy, balls trajectories and probability distribution of velocities, in both the normal and tangential directions. The experimental validation was supported by X-ray Powder Diffraction Line Profile Analysis (XRPD-LPA) of ground calcium fluoride (CaF_2).

2. Methods

Planetary ball mills consist of two or more jars rotating both around its own axis and the line of symmetry of the supporting plate (radius $R_P = 125$ mm, see Fig. 1). The motion of the milling media (balls) inside the vial, which is driven by the resulting field of two centrifugal forces and the Coriolis force, causes impacts that transfer compressive and shear forces to the powder charge. These forces lead to structural and microstructural alterations and/or mechanochemical effects [13,14].

As shown by Burgio et al. [15], at optimum milling conditions the trajectory ideal for a single ball follows a point on the vial circumference until the resulting field of forces acts to carry the ball across the jar to the opposite point, perpendicularly to the jar surface. In a real scenario, trajectories depend on the interactions between many balls and forces are exchanged in every possible direction with respect to the impact reference frame. To enhance the normal component of the transferred force, a half-moon shaped jar (HM) is proposed, with a design based on a flat surface halfway between the original curved wall and the vial axis. This way, when balls rolling along jar wall approach the flat surface, they are forced to follow the *ideal path*, therefore increasing the amount of energy along impact axis and thus causing a more intense comminution effect.

2.1. Computer simulations

Computer simulations are a useful tool to explore thoroughly and characterize the milling process, providing insights into kinematic and dynamic quantities, mostly inaccessible to experiments. In this study, the Fritsch Pulverisette 4 (P4 [16]) planetary ball mill, equipped with either the Cylindrical (CY) or the HM jar, was modelled using the multibody dynamics software MSC.Adams [17], as reported in [11]. Twelve steel balls (radius 6 mm) were placed randomly inside each jar and angular velocities ω and Ω were applied to the two cylindrical

Table 1
Physical properties of CY, HM jars and milling media for the presented case study.

CY, HM jars (AISI 304)		Spheres (AISI C1020)	
Density	8.03 g/cm^3	Density	7.85 g/cm^3
Young modulus	193 GPa	Young modulus	200 GPa
Poisson ratio	0.29	Poisson ratio	0.29