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Parameter Optimization of Wet Stirred Media Milling Using an Intelligent Algorithm-Based Stressing Model

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Abstract

Wet stirred media milling (WSMM) is a popular grinding method used to produce important ultrafine-particle materials, such as pigments, pharmaceuticals, and pesticides. Therefore, it is crucial to improve the process capability and quality of WSMM by setting optimal parameters. This study proposes a multi-objective optimization methodology based on an intelligent algorithm to optimize the ultra-fine grinding parameters; this can mitigate the issue whereby grinding parameters are difficult to determine during wet grinding industrial production. A mechanistic model is proposed based on the analysis of energy dissipation mechanisms. The specific energy in the WSMM process is quantified using a stressing model. A shuffled frog leaping algorithm (SFLA)-based stressing model is proposed to maximize the specific stress intensity and specific stress number of the entire system under the constraint of the product particle size and grinding time, which provides the optimal process parameters. The performance of the proposed strategy is validated using two case studies in different industrial optimization scenarios. The result of the first case study illustrates that, in comparison to a quadratic programming-based response surface methodology, the proposed SFLA-based stressing model greatly enhances the wet grinding efficiency (decreasing P_{80} from 3.28 μm to 2.88 μm). In the second case study, the parameter optimization under different feed particle sizes and different productivities was discussed. The results confirmed that the optimized parameters can achieve the minimum particle size (P_{50} = 1.78 μm) and maximum solid concentration (C_v = 120 g/L) within the minimum grinding time (t_g = 5 min). The contribution of our work lies in the fact that the proposed SFLA-based stressing model can direct multiple-objective decision-making in a more efficient way without requiring costly experimental procedures to acquire the optimized parameters in WSMM. The proposed approach is systematic and robust and can be integrated into WSMM architectures for parameter optimization in other complex wet grinding systems.

Keywords: parameter optimization; shuffled frog leaping algorithm; stressing mode; wet stirred media milling

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1. Introduction

Wet stirred media milling (WSMM) has become an established grinding technique for producing fine and ultrafine materials, such as foods, pharmaceutical products, pesticides, ceramics, and other materials [1,2]. Due to the very high stress intensity and number of

stress events per unit time, energy is more concentrated in particle comminution, reducing ineffective energy consumption. Benefiting from the efficient concentration of energy, the specific energy consumption of WSMM is lower than those of tumbling and vibrating ball mills when producing micron-, sub-micron-, and even nano-scale particles [3]. Therefore, WSMM is a robust, reproducible, scalable, and environmentally friendly mechanized physical particle comminution process. WSMM is a complex process that is regarded as a "black box" [4]. It depends on several process variables, which can be divided into two categories: (1) mill parameters, such as the stirrer tip speed, mill volume, grinding media diameter, and density/filling ratio; (2) suspension properties, such as the solid volume concentration, viscosity, shear rate, flow rate, and particle size/Young's modulus. In general, the diverse product properties and variety of available equipment make the process variables difficult to ascertain. Therefore, numerous parameters and uncontrollable factors make it challenging to optimize WSMM parameters [4].

Parameter optimization involves two aspects: (1) identifying the constraints of WSMM and (2) adjusting the input variables to obtain the desired performance. Many studies have attempted to identify the most important WSMM parameters and characterize and optimize the milling process. Kwade et al., Alamprese et al., and Hou et al. optimized WSMM parameters using a stressing model, response surface methodology (RSM), and a combined RSM-genetic algorithm (GA) approach, respectively [5–8]. These studies verified the feasibility of model-driven optimization; however, the former relied on extensive grinding tests to determine key parameters, while the latter two showed limited adaptability in multi-objective coupling scenarios. Based on a three-level Box–Behnken design, Celep et al. employed an RSM with quadratic programming (QP) for the modeling and optimization of process parameters during the ultra-fine grinding of refractory Au/Ag ores [9]. A second-order regression model was fitted to experimental data and then optimized using the quadratic programming method to minimize the d80 size within the experimental range studied. Mäntynen et al. proposed a three-step optimization procedure for the production of colorant paste. First, a Plackett–Burman experimental design was used to determine the most significant grinding variables. Second, quadratic models of tinting strength and grinding power were constructed, and finally, a set of Pareto-optimal solutions was obtained based on the lower and upper limits of the corresponding grinding conditions [10].

Using an enhanced stress model, Breitung-Faes et al. predicted the optimum parameters for inorganic nanoparticle production by WSMM. By analyzing the relationships between the average optimum stress energies and single-particle-breakage variables of different product materials, an optimum set of parameters can be obtained. However, comparative analyses of grinding capabilities under optimal parameters remain lacking [11]. Using residence time distribution modeling, Montalescot et al. studied the hydrodynamics involved in WSMM. Stress intensity–stress number (SI–SN) modeling has been successfully applied to microalgae production, with its operating parameters optimized to minimize energy consumption [12]. Based on a three-factor, three-level, and face-centered CCD, Nekkanti et al. studied the effects of bead milling process variables on the manufacturing process of drug nanoparticles. The variables investigated were the motor speed, pump speed, and bead volume, and the responses were the milling time, particle size, and process yield. The optimum process was obtained using Design-Expert ® (version. 7.3.1; Stat-Ease Inc., Minneapolis, MN) program to evaluate both the repeatability and reproducibility of the bead milling technique [13]. Flach et al. evaluated the characteristic parameters of a stress model and a micro-hydrodynamic model for the process optimization of a nanosuspension prepared by WSMM. They showed that process optimization resulted in the same optimum set of operating parameters. However, comparative analyses of the influence of optimized parameters on the mill grinding capacity are lacking [4]. Brunaugh et al. argued that micronization, such as that achieved by

stirred media milling, is well-suited to parameter optimization, due to its generally low-yield high-energy consumption process. They reviewed the optimization of pharmaceutical milling parameters through design space development, theoretical and empirical modeling, and the monitoring of critical quality attributes [14]. Katircioglu-Bayel. studied the effects of operating parameters on the product particle size, surface area, and energy consumption. The optimum process parameters were determined through numerical calculation and correlation analysis [15]. Santosh et al. studied the effects of the stirrer speed, grinding time, feed size, and solid concentration on the product size and energy consumption when grinding the chromite ore-bearing platinum group of elements in a laboratory stirred mill. Two regression process models were developed and optimized using quadratic programming and Design Expert software [16]. Katircioglu-Baye et al. investigated the impact of four factors (the stirrer speed, grinding time, media filling ratio, and solid mass fraction) on the product particle size (d50). By using the three-level Box–Behnken design, the optimum operating parameters for minimizing the d50 size were determined [17]. Amini et al. proposed a response surface methodology (RSM) to determine the optimum parameters for extracting phenolics from pomegranate peel [18]. Guner et al. studied the influence of bead sizes and bead mixtures on breakage kinetics during the nanomilling of drug (griseofulvin) suspensions. The experimental results show that the use of bead mixtures did not lead to notable process improvements, and 100 μm beads outperformed bead mixtures with the advantages of fast breakage, low power consumption, and heat generation [19]. Wang et al. established the relationship between the grinding power consumption and operating parameters in a tower mill based on the BP neural network optimized by the GA to obtain the optimum conditions [20]. BAHETI et al. used canonical analysis with the help of SYSTAT software (https://www.grafiti.com) to optimize the model equation and determine the optimum conditions for operating parameters of high-energy planetary ball mills [21]. Gbadeyan et al. analyzed the signal-to-noise (S/N) ratio to determine the optimum process parameters in nano calcium carbonate production [22]. Guo et al. used response surface methodology (RSM) to optimize the three key process parameters based on the grinding technology efficiency as the evaluation criterion. They found that the order of significance of the parameters affecting the grinding efficiency was as follows: grinding time > media filling rate > stirrer speed [23].

Table 1 provides an overview of current research on the topic.

From the abovementioned literature, we identified three main issues in implementing WSMM parameter optimization.

(1) The goal of parameter optimization is to understand the fundamental behavior of WSMM. Currently, WSMM parameter optimization relies heavily on empirical approaches, experimental design, and modeling based on the first process [16]. However, WSMM is expensive due to its low mill capacity and high energy consumption [24]. Therefore, reducing the experimental cost and improving the optimization results remain challenging.

(2) Parameter optimization in WSMM usually involves multi-objective optimization (MOO) to characterize the product quality in response to variations in the input. Stress models and RSM are currently the most widely used parameter optimization strategies. However, these models rely on experience or theoretical analysis, while optimization analysis depends heavily on experimental data [6]. Due to the diversity of product quality attributes, which are sometimes contradictory and conflicting, stress models and RSMs need further development for use in MOO.

(3) Few studies in this field have used intelligent algorithms. These are metaheuristic algorithms, which generally do not require the continuity and convexity of the objective function and constraints. They have high adaptability to data uncertainty and are regarded as potentially powerful tools for solving MOO problems [25]. However, their application to WSMM parameter optimization requires further study.

Table 1. Literature summary on process optimization in WSMM.

Process Parameters	Mill Type	Optimization Approaches	Object/Response	Ref.
Stirrer tip speed Grinding media size	Lab scale disc mill	Stress model Micro hydrodynamic model	Particle sizes Specific energy input	[4,14]
Stirrer tip speed Grinding media size Grinding media density	Lab scale disc mill	Stress model	Average stress energy Specific energy input	[5,6,11]
Media filling ratio Grinding media size Stirrer speed Grinding time Feed size Solid concentration	Vertical-type stirred media mill	Box–Behnken statistical design	Product particle size Surface area Energy consumption	[15,16]
Bead size Filling ratio Flow rate	Dyno®-mill	Stress intensities and stress number	Cell destruction kinetics Specific energy	[12]
Rotational speed Solid-to-solvent ratio Ethanol-to-water ratio	Micro wet milling	Response surface methodology	Total phenolic content Total anthocyanin content	[18]
Refining time Agitator shaft speed Bead volume	Stirred media mill	Response surface regression Central composite design	Electricity consumption Particle size Iron content Refining time Process yield	[7,13]
Tinting paste viscosity Bead charge Grinding bead size Rotor velocity Recycle flow rate	DCP-UPERFLOW® high-performance agitated media mill	Pareto-optimal solutions	Tinting strength Grinding power	[10]
Milling time Flow velocity Agitator rotation Weight ratio Bead filling ratio	Vertical milling machine	Taguchi method Response surface method Genetic algorithm	Mean grain size Variance of grain size	[8,9]
Zirconia bead sizes Bead loading	Wet stirred media milling	Microhydrodynamic model augmented with a decision tree	Solution flow rate Gas flow rate Solution concentration	[19]
Stirrer speed Grinding time Media filling ratio Solid mass fraction	Stirred media mill	Three-level Box–Behnken design	Mean grain size	[17]
Grinding concentration Screw speed Medium filling rate Material–ball ratio	Tower mill	BP neural network optimized by the GA	Grinding power consumption	[20]
Milling time Milling speed Ball to material ratio ball size	High energy planetary ball mill	A three-level Box-Behnken design combining a response surface methodology	Particle size and sticking of material to mill	[21]
Microparticle loading Number of runs	Supermasscolloider wet milling	Response surface design method	Particle size	[22]

The goal of this study is to address these three issues. To achieve this, a parameter optimization methodology is proposed based on a stressing model and intelligent algorithm.

2. Materials and Methods

2.1. Materials

Heavy calcium carbonate (HCC), which is widely used in daily chemical industries such as rubber, plastics, papermaking, paints, inks, cables, and building supplies, is employed as an experimental material in the second case study. The specific gravity of the HCC was determined to be 2.80 g/cm^3. The HCC sample was ground in laboratory rod mill at 50–120 g/L pulp density with different mono-sized fractions (50% passing size, D50 = 6–32 μm) and riffled to obtain 60–130 g representative sub-samples prior to ultrafine grinding using the pin-type horizontal stirred mill (RTSM-05, Anhui Root Intelligent Equipment Co., Ltd., Suzhou City, Anhui Province, China). The earlier chemical and mineralogical analysis studies carried out on HCC samples are shown in Table 2.

Table 2. Analysis of HCC elements in samples.

Element	$CaCO_2$	MgO	Al_2O_3	Fe_2O_3	Insoluble Matter	Loss on Ignition
Content (%)	98.5	0.8	0.1	0.05	0.2	0.35

On the other hand, an antimonial refractory gold/silver ore sample was obtained from Akoluk (Ordu, Turkey) and used as a grinding material. It was determined to be rich in silver, containing 220 g/ton Ag and 20 g/ton Au. Its main chemical composition is shown in Table 3 [9]. The material was ground at a 50% pulp density for P80 = 60 μm prior to ultrafine grinding using a wet stirred mill [9].

Table 3. Chemical composition of the grinding material [9].

Element	SiO_2	Fe_2O_3	Cu	Ba	Sb	Zn	Sr	Tot. S	Pb	Tot. C	Loss on Ignition
Content (%)	52.15	1.28	0.04	17.10	1.64	1.50	0.31	6.89	0.43	0.05	4.60

2.2. Methods

2.2.1. Energy Dissipation Mechanism in WSMM

In actual comminution processes, the performance of WSMM is contingent on the total energy transferred to the product particles. Though there are many different types, geometries, and sizes of products made by WSMM in various industries, the operating principle remains similar: a motor drives a stirrer, which stirs a suspension. Energy is transferred from the suspension to the grinding medium, and the dominant particle breakage mechanism is shear and friction between grinding media, rather than normal impact—this is particularly evident in the ultra-fine grinding of materials [26]. To analyze the mechanical energy absorbed by particles, it is essential to analyze the energy dissipation mechanism of the energy transfer process. Figure 1 provides a schematic overview of the energy dissipation mechanisms of WSMM.

Energy transfer and dissipation among the various components of WSMM processes, such as the input shaft, stirrer, suspension, grinding media, and product particles, can be regarded as largely linear continuous processes, although coupling exists locally. In terms of particle stress, certain coupling effects can be ignored, for example, the dynamic effect of the grinding media on the suspension. The comminution result is determined by the energy transferred from the grinding media to the product particles [5]. However, only a small part of the input energy of the chamber is used for particle stressing [5]. Figure 1 shows

that the energy introduced to the chamber experiences a large amount of dissipation before reaching the product particles. The energy dissipation process is illustrated in Figure 2, in which blue arrows represent the direction of energy transfer.

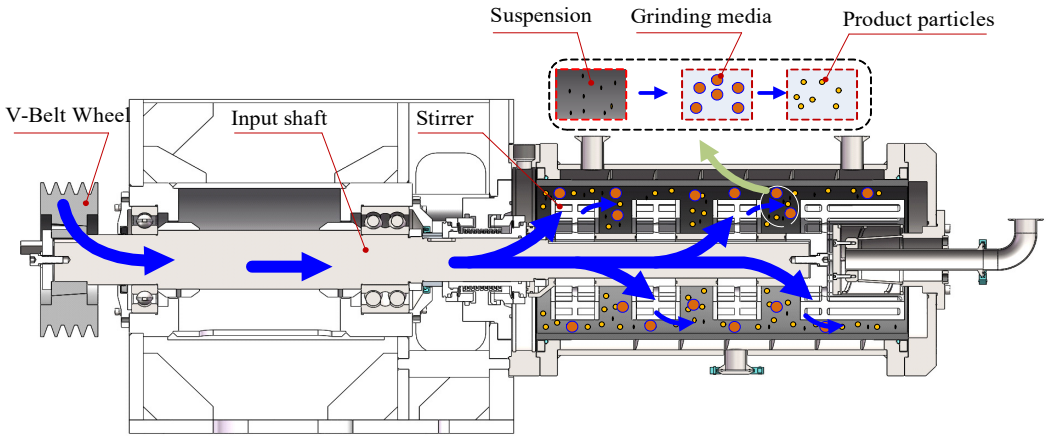


Figure 1. Schematic overview of energy dissipation in WSMM.

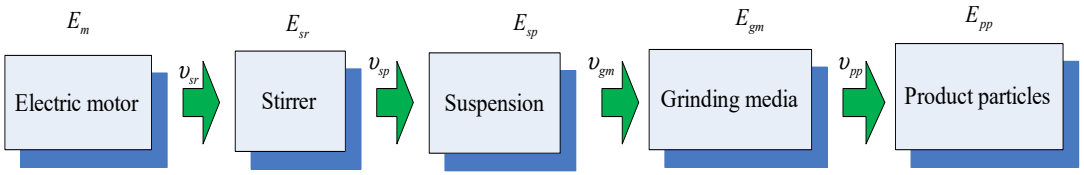


Figure 2. Energy transfer and dissipation process.

In Figure 2, E_m is the specific energy provided by the electric motor, which transfers energy to the stirrer via the V-belt wheel and input shaft, as shown in Figure 1. v_{sr} is the energy transfer coefficient, which is the percentage of kinetic energy transferred from the electric motor E_m and consumed by the stirrer E_{sr}. v_{sr} can be estimated according to Equation (1).

$$\begin{aligned} v_{sr} &= \frac{E_{sr}}{E_m} \\ &= \frac{\int_0^t P_0 d_t}{\int_0^t P d_t} \end{aligned} \tag{1}$$

Here, P and P_0 represent the electric motor power and the power consumed by the stirrer, respectively. The stirrer also outputs energy to the suspension. The energy consumed by the suspension, E_{sp}, is expressed as Equation (2).

$$E_{sp} = E_m(1 - v_{sr})v_{sp}, \tag{2}$$

where v_{sp} is the energy transfer factor of the suspension. The energy is dissipated by liquid friction and facilitates dispersion but not particle stress. The energy is related to the viscosity, shear rate, and volume fraction of the suspension and is dissipated as heat and vibration due to shear friction inside the suspension [5]. Like the stirrer, the suspension also transfers part of the energy to the grinding media via liquid–grinding media, viscous friction, and lubrication. This energy component is also divided into two parts: the non-particle-stressing energy E_{gm} and particle-stressing energy E_{pp}. Similar to Equation (2), if the energy transfer factor v_{gm} of the beads is given, E_{gm} can be calculated using Equation (3).

$$E_{gm} = E_m(1 - v_{sr})(1 - v_{sp})v_{gm} \tag{3}$$

The non-particle-stressing energy E_{gm} is dissipated as heat and vibration in two main ways: (1) The suspension is displaced by approaching grinding media, wherein the kinetic energy is reduced due to viscous damping by the suspension. (2) The grinding medium contacts product particles without stressing them [4]. The particle-stressing energy E_{pp}, which is the energy used in particle crushing, is mainly manifested by grinding media wear and deformation, and is a function of the particle–grinding medium elasticity ratio [27]. Similar to Equation (3), E_{pp} is calculated according to Equation (4).

$$\begin{aligned} E_{pp} &= E_m(1-v_{sr})(1-v_{sp})(1-v_{gm})v_{pp} \\ &= v_e E_m \end{aligned} \tag{4}$$

Here, v_e is the energy transfer factor, and E_{pp} is the specific energy delivered to product particles. If the ith stress intensity SI_i and the frequency distribution SF_i of all stress events are given, then E_{pp} can be written as

$$\begin{aligned} E_{pp} &= \frac{\sum_{i=1}^{n} SI_i \cdot SF_i}{m_p} \\ &= \frac{\overline{SI}_p \cdot \sum_{i=1}^{n} SF_i}{m_p} \\ &= \frac{\overline{SI}_p \cdot SN_p}{m_p} \end{aligned}, \tag{5}$$

where $\overline{SI}_p$ and SN_p are the average stress intensity and total stress number of product particles, respectively.

2.2.2. Parameter Optimization Using the Stressing Model

The distribution of the stress intensity will not change as long as the geometry of the chamber remains unchanged. By taking into account the elastic modulus of the grinding media and product particle materials [27], the stress intensity of the grinding media in a single stress event, SI_{gm}, can be defined as follows:

$$\begin{aligned} SI_{gm} &= d_{gm}^3 \cdot v_{gm}^2 \cdot \rho_{gm}\left(1+\frac{y_p}{y_{gm}}\right)^{-1} \\ &= \pi^2 \cdot d_{gm}^3 \cdot n_{sr}^2 \cdot D_{sr}^2 \cdot \rho_{gm}\left(1+\frac{y_p}{y_{gm}}\right)^{-1} \end{aligned}, \tag{6}$$

where d_{gm}, n_{sr}, D_{sr}, and ρ_{gm} are the diameter of the grinding beads, stirrer speed, stirrer diameter, and grinding media density, respectively.y_p and y_{gm} are the Young's moduli of the product and grinding media, respectively. Since the linear velocities of the grinding media in the chamber are different, so is the stress intensity. Introducing the stress intensity factor δ_I, the average stress intensity of the product particles, $\overline{SI}_p$, is determined as

$$\overline{SI}_p = \delta_I \cdot SI_{gm}. \tag{7}$$

In addition to the stress intensity, the stress number SN_{gm} denotes the number of stress events, and is defined as follows [28].

$$\begin{aligned} SN_{gm} &= n_{sr} \cdot N_{gm} \cdot t_g \\ &= n_{sr} \cdot \frac{V_{gc} \cdot \varphi_{gm} \cdot (1-\phi)}{\frac{\pi}{6} \cdot d_{gm}^3} \cdot t_g \end{aligned}, \tag{8}$$

where n_{sr}, V_{gc}, φ_{gm}, ϕ, and t_g are the stirrer rotation speed, effective volume of the chamber, grinding media filling level, porosity, and grinding time, respectively. In a multi-pass

continuous grinding process, if the total volume of the suspension to be ground is V_Q, and the flow rate of the suspension is v_c, the grinding time t_g can be derived as follows:

$$t_g = k \cdot \frac{V_Q}{v_c}, \tag{9}$$

where k is the number of times the suspension has been ground. Since not all grinding media can capture product particles in every stress event, given the stress number factor δ_N, the total stress number that the grinding media can capture the product particles, SN_p, can be determined as follows.

$$SN_p = \delta_N \cdot SN_{gm} \tag{10}$$

Combining Equations (5)–(8), the specific energy E_{pp} can be derived:

$$\begin{aligned} E_{pp} &= \frac{\overline{SI}_p \cdot SN_p}{m_p} \\ &= \frac{\delta_I \cdot SI_{gm} \cdot \delta_N \cdot SN_{gm}}{m_p} \\ &= \delta_S \cdot \frac{SI_{gm} \cdot SN_{gm}}{m_p} \end{aligned}, \tag{11}$$

where δ_S is the total energy transfer coefficient. Since more than one particle is captured by the grinding media in each stress event, it is necessary to calculate the stress intensity and stress number of a single particle. Figure 3 shows a scenario where two product particles are stressed by two grinding media. In the actual grinding process, the number of product particles captured depends on the active volume between two grinding media in a stress event [29]. This active volume is proportional to the diameter of the grinding media and that of the product particles.

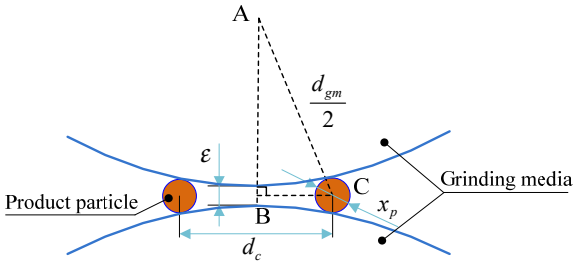


Figure 3. Active volume calculation.

Figure 3 shows the spatial relationship between grinding media and product particles. The orange circles denote product particles, which have a diameter of x_p. The minimum distance between the grinding media is ε, and d_c is the diameter of the active volume. According to the triangle ABC, d_c can be calculated as

$$d_c = \left\{ \left(x_p - \varepsilon \right) \left(x_p + \varepsilon + 2d_{gm} \right) \right\}^{1/2}, \tag{12}$$

when $\varepsilon = 0$, and x_p and d_{gm} are constants. d_c takes the maximum value of $d_{c,max} = \left(x_p^2 + 2x_p d_{gm} \right)^{1/2}$. The active volume V_c can be derived as follows:

$$\begin{aligned} V_c &= \pi \cdot \frac{d_{c,max}^2}{4} \cdot x_p \\ &= \pi \cdot \frac{\left(x_p^2 + 2x_p d_{gm} \right)}{4} \cdot x_p \\ &= \frac{\pi}{4} \left(x_p^3 + 2x_p^2 d_{gm} \right) \end{aligned}. \tag{13}$$

If the solid concentration of product particles C_v is given, then the stress intensity per unit product quality $\overline{SI}_{p,s}$ in a stress event can be estimated according to Equation (14).

$$\begin{aligned} \overline{SI}_{p,s} &= \frac{\overline{SI}_p}{V_c \cdot C_v} \\ &= \frac{\delta_I \cdot SI_{gm}}{\frac{\pi}{4}\left(x_p^3 + 2x_p^2 d_{gm}\right) \cdot C_v} \end{aligned} \tag{14}$$

Similarly, the number of stress events per unit product quality, $SN_{p,s}$, can be found using the following equation:

$$\begin{aligned} SN_{p,s} &= \frac{SN_p}{m_p} \\ &= \frac{\delta_N \cdot SN_{gm}}{V_c\left(1-\varphi_{gm}(1-\phi)\right) \cdot C_v} \end{aligned}, \tag{15}$$

where m_p is the mass of product particles in the chamber, and V_c is the volume of the chamber. To improve the grinding efficiency, it is necessary to increase the stress number $SN_{p,s}$ per unit time and the stress intensity $\overline{SI}_{p,s}$. Further, the product particle diameter x_p must be limited to a certain range, x_m^*. Mathematically, this can be expressed as

$$\begin{aligned} &Max: SN_{p,s} = \frac{6 \cdot \delta_N \cdot n_{sr} \cdot \varphi_{gm}(1-\phi) \cdot \gamma}{\pi \cdot C_v \cdot \left(1-\varphi_{gm} \cdot (1-\phi)\right) \cdot d_{gm}^3} \cdot \left(\frac{\alpha v_c}{1+v_c^k} + 1\right) \\ &Max: \overline{SI}_{p,s} = \frac{4\pi \cdot \delta_I \cdot d_{gm}^3 \cdot n_{sr}^2 \cdot D_{sr}^2 \cdot \rho_{gm}}{C_v \cdot \left(x_p^3 + 2x_p^2 \cdot d_{gm}\right)} \cdot \left(1 + \frac{y_p}{y_{gm}}\right)^{-1} \\ &Subject to: x_p \le x_m^* \end{aligned}, \tag{16}$$

where v_c is the slurry flow rate. γ, α, and k represent slurry flow rate coefficients.

2.2.3. Optimization Strategy Using the Intelligent Algorithm

Process optimization problems are actually multi-objective optimization (MOO) problems. Under MOO conditions, the optimized process parameter set is, in fact, a Pareto-optimal set. Conventional mathematical programming (MP) is generally employed to solve such problems, which is sensitive to the Pareto-optimal frontier and requires the objective function and constraints to be differentiable [30]. In contrast, the meta-heuristic algorithm is not subject to such restrictions and is regarded as a powerful tool for solving MOO problems [31]. Therefore, a meta-heuristic algorithm—the shuffled frog leaping algorithm (SFLA)—is developed for process optimization in WSMM. The SFLA combines the merits of shuffled complex evolution (SCE) and particle swarm optimization (PSO). Therefore, in comparison to the GA or ACO algorithm, the SFLA has better global and local search capabilities. Figure 4 shows the process optimization flowchart using the SFLA; the right side shows the SFLA optimization process and involved parameters.

The procedures are presented as follows. Based on the stressing model, the mechanism of energy transfer and dissipation in the WSMM is determined. Subsequently, the objectives and constraints for process optimization are proposed. The SFLA is employed for multi-objective optimization. The SFLA is designed as a meta-heuristic algorithm for seeking a solution to a combinatorial optimization problem by using a fitness function. A virtual frog acts as the host of the optimized parameter set $\{n_{sr}, \varphi_{gm}, d_{gm}, x_p, C_v, v_c\}$, where each parameter is treated as a meme for cultural evolution. First, the virtual frog population is randomly generated within the constraints. Each frog has a fitness value that measures the goodness of the individual. The virtual frog population is partitioned into several memeplexes, and roulette is used to construct sub-memeplexes. Secondly, the worst frog in the sub-memeplexes is improved by the best frog in the sub-memeplexes and the global optimal frog, which completes the local search and global information exchange processes. The convergence criteria are met if one frog carries the "best memetic pattern so far" for

fifteen consecutive instances. Finally, the optimized parameters are verified by a wet grinding test to further optimize the actual process.

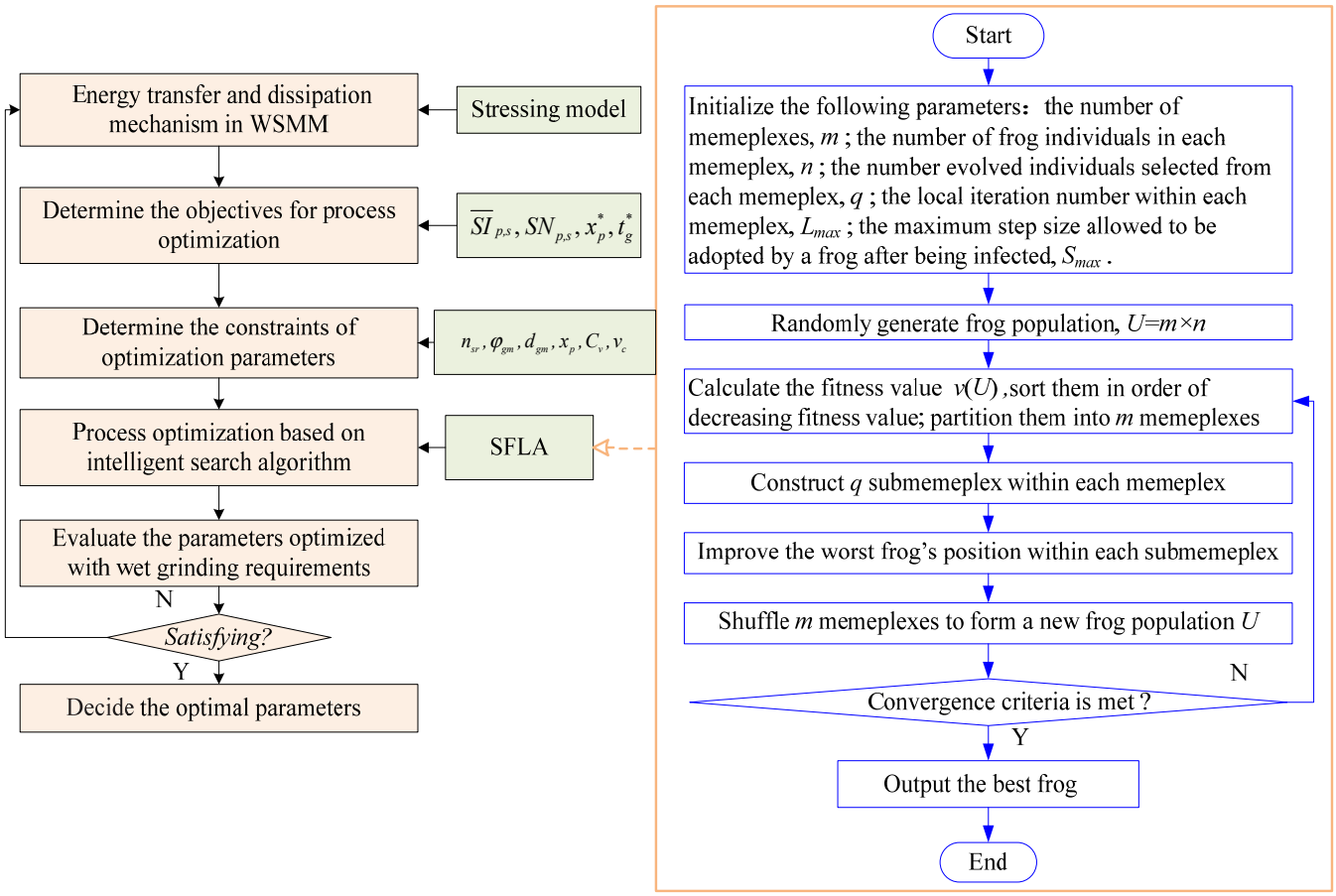


Figure 4. SFLA-based stressing model methodology for parameter optimization.

3. Results

In order to illustrate the proposed parameter optimization approach on the WSMM system, two case studies, one for vertical wet grinding and the other for horizontal wet grinding, were developed.

3.1. Application on Pin-Type Vertical Stirred Mill

An antimonial refractory gold/silver ore sample was used as a grinding material. The material pretreatment and its material characteristics are described in Section 2.1. Grinding tests were carried out in a pin-type vertical stirred mill (Figure 5).

The stirrer consisted of 14 pins fitted to a shaft (Ø8.9 mm diameter). The chamber volume was 580 cm^3, and the pulp density was maintained at 35% w/w during the grinding process. Three stirrer speeds were used: 250, 500, and 750 rpm. The grinding medium was stainless steel beads with an average density of 7.68 g/cm^3 and three diameters (1, 3, 5 mm). The bead load was controlled at three charge ratios (60, 70, 80%). The coded level of the ball diameter (v_1), stirrer speed (v_2), and ball charge ratio (v_3) can be obtained using the following formula:

$$\begin{aligned} v_1 &= \frac{d_{gm}-3}{2} \\ v_2 &= \frac{n_{sr}-500}{250} \\ v_3 &= \frac{\varphi_{gm}-70}{10} \end{aligned} . \quad (17)$$

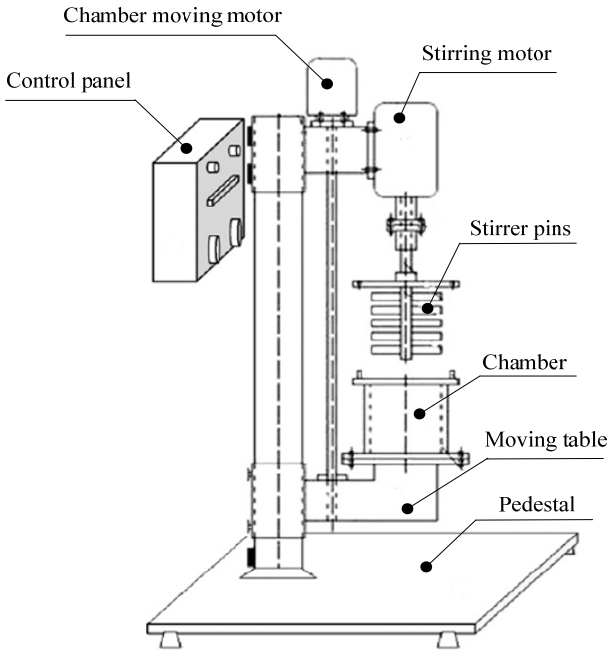


Figure 5. Stirred media mill [9].

The coded values and actual values in each run are listed on the left side of Table 4. The particle size was determined using a Malvern Mastersizer 2000 (Malvern Panalytical, Malvern, Worcestershire, UK), with the mean of five replicates recorded.

Table 4. Grinding process parameters and predicted results as a reference benchmark [9].

Run	Coded Level			Actual Level			P_{80} (μm)	
	v_1	v_2	v_3	d_{gm}/mm	n_{sr}/rpm	φ_{gm}/%	Observed	Predicted
1	−1	1	0	1	750	70	4.35	4.32
2	0	−1	−1	3	250	60	9.23	9.26
3	0	0	0	3	500	70	5.61	5.73
4	0	0	0	3	500	70	5.75	5.73
5	−1	−1	0	1	250	70	9.79	9.86
6	0	1	−1	3	750	60	5.17	5.08
7	1	0	1	5	500	80	8.05	7.66
8	−1	0	−1	1	500	60	6.80	6.78
9	−1	0	1	1	500	80	5.22	4.82
10	0	−1	1	3	250	80	7.68	8.68
11	1	−1	0	5	250	70	10.41	11.00
12	1	0	−1	5	500	60	7.92	7.90
13	0	0	0	3	500	70	5.83	5.73
14	1	1	0	5	750	70	6.66	7.14
15	0	1	1	3	750	80	4.64	3.46

The second-order model representing P_{80} was expressed as a function of the ball diameter (v_1), stirrer speed (v_2), and ball charge ratio (v_3) for coded units based on the experimental data. To compare the effects of process optimization, we set the grinding time t_g = 10 min. Then, the equation can be expressed as [9].

$$P_{80} = 5.73 + 0.99v_1 - 2.35v_2 - 0.55v_3 + 1.26v_1^2 + 1.09v_2^2 - 0.20v_3^2 + 0.42v_1v_2 + 0.43v_1v_3 - 0.26v_2v_3 \quad (18)$$

Table 4 shows the measured and predicted values of the product particle diameter P_{80}. Each run was performed in duplicate, and their mean values were calculated. The

predicted values were obtained from Equation (18). Using the data in Table 4, the mean relative error of the predicted values ($MRE(P_{80})$) was found to be 0.048, which indicates that Equation (18) is reliable and has a high predictive accuracy.

As illustrated in Equation (16), the process optimization had the multiple objectives of stress number $SN_{p,s}$ (primary) and stress intensity $\overline{SI}_{p,s}$ (secondary) under the constraints of particle diameter x_m^* and grinding time t_m^*. This is because, in micro- and nano-material grinding, the main mechanism of particle crushing is shear and friction between grinding media, rather than normal impact [32]. Equation (16) was optimized using SFLA to minimize P_{80} within the experimental range. The SFLA algorithm was implemented in the mathematical software package (MATLAB 2022b). The SFLA parameters were set as follows: number of memeplexes m = 30, number of frogs in each memeplex n = 50, number of evolved individuals selected from each memeplex q = 50, local iteration number within each memeplex L_{max} = 30, and maximum step size to be adopted by a frog after being infected S_{max} = 1. To facilitate industrial applications, the "actual levels" in Table 4 were employed as the optimized process parameters. The optimal levels of the variables were found to be the ball diameter = 1 mm, ball charge ratio = 80%, and stirrer speed = 750 rpm, with a prediction of P_{80} = 2.88 μm. In comparison to the actual levels listed in Table 4, the optimized process parameters can achieve the minimum product particle size within the grinding time.

3.2. Application on Pin-Type Horizontal Stirred Mill

Heavy calcium carbonate (HCC) is used as an experimental material. The material pretreatment and its material characteristics are described in Section 2.1. Ultra-fine grinding tests are carried out in a batch scale pin-type horizontal stirred mill (RTSM-05) provided by Anhui Root Intelligent Equipment Co., Ltd. (Suzhou City, Anhui Province, China) (Figure 6). The stirred mill is comprised mainly of a grinding chamber, stirrer, motor, control box, diaphragm pump, and water cooler. The grinding chamber, which is jacketed for cooling water circulation, has a volume of 0.5 L. The maximum power of the motor is 4 kW, and the stirrer speed can be varied from 600 to 1300 rpm. The yttrium-stabilized zirconia beads, whose average density is 6.07 g/cm^3 with a size of d_{gm} = 0.30 mm, are used as grinding medium. The bead load for all the tests is fixed at 60% of the net mill volume. The product size distribution analysis was carried out by the laser diffraction method using a BT-2003 provided by Dandong Better size Instrument Co., Ltd. (Dandong City, Liaoning Province, China), which is China's No. 1 player in the particle sizing business. Each particle size analysis was performed in three replicates, and the mean is presented in the results.

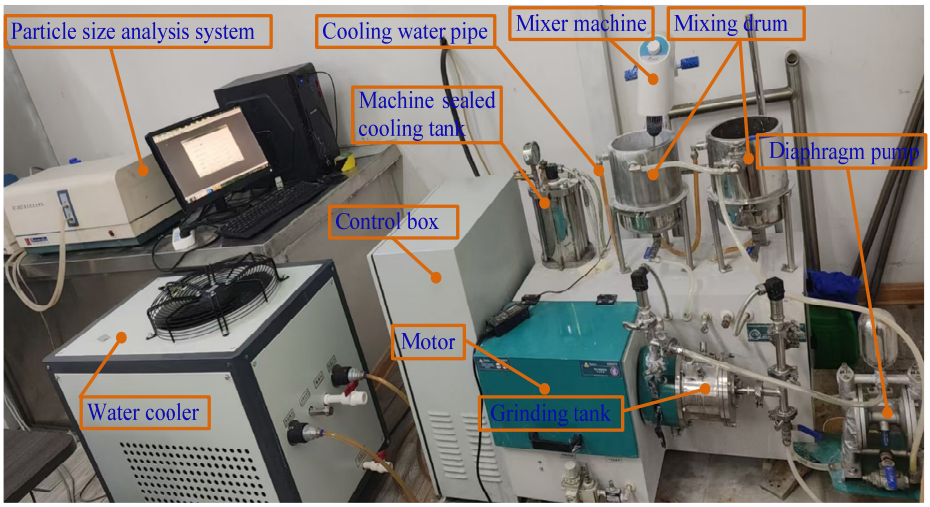


Figure 6. Isometric view of RTSM-05 horizontal stirred mill.

A total of 50 experiments, in which the process parameters were provided and validated by company technicians, were conducted to assess the effect of the stirrer speed (n_{sr}), grinding time (t_g), initial product particle size (x_p), and solids concentration (C_v) on the product particle size (P_{50}). The range of the variables and the observed product size (P_{50}) considered in this work are listed in Table 5.

Table 5. Grinding process parameters and the observed product size (P_{50}).

No.	Actual Level of Variables				Observed Results
	n_{sr}/rpm	t_g/min	x_p/μm	C_v/g/L	P_{50}/μm
1	1200	9	16	90	3.19
2	700	7	20	120	16.82
3	800	9	8	110	4.24
4	700	17	10	60	3.66
5	1300	9	17	60	2.55
6	700	5	22	80	17.17
7	1200	7	17	50	3.56
8	900	13	32	90	5.30
9	800	11	32	80	9.58
10	1300	5	16	100	4.36
11	1000	15	32	100	2.66
12	800	13	22	120	6.84
13	800	21	14	90	3.75
14	1100	9	14	120	4.12
15	1000	11	22	110	5.44
16	1000	17	16	70	2.04
17	1300	11	10	120	1.85
18	800	15	20	70	3.91
19	1000	5	10	90	2.66
20	800	19	16	50	3.32
21	1200	21	22	70	2.55
22	1200	13	11	100	2.13
23	1200	15	10	110	1.98
24	900	11	20	50	4.40
25	1000	19	14	80	2.14
26	1000	21	17	120	2.46
27	900	9	10	80	3.85
28	1100	19	22	60	1.85
29	700	19	8	100	2.83
30	700	21	11	110	4.34
31	1100	5	17	70	5.97
32	1100	21	20	100	2.41
33	900	21	16	60	2.36
34	800	5	11	60	3.98
35	900	19	17	110	3.56
36	1100	11	11	90	2.60
37	1300	17	22	50	3.40
38	700	9	32	70	11.69
39	900	17	14	100	2.63
40	700	13	14	50	4.48
41	900	5	8	120	3.67
42	1000	13	20	60	4.43
43	1200	17	20	80	3.55
44	1200	11	8	60	2.18
45	900	7	11	70	3.79
46	1100	7	16	80	2.88
47	1300	7	14	110	2.76
48	700	15	17	90	9.52
49	1000	9	11	50	2.34
50	800	7	10	100	5.56

SFLA-based stressing model methodology is employed to acquire the optimal process parameters. In order to ensure the product productivity, the solids concentration, C_v, should be ensured to take the maximum value. The SFLA algorithm was implemented in the mathematical software package (MATLAB 2022b), with the following parameters: number of memeplexes m =30, number of frogs in each memeplex n =45, number of evolved individuals selected from each memeplex q = 30, local iteration number within each memeplex L_{max} = 25, and maximum step size to be adopted by a frog after being infected S_{max} = 1. The optimal levels of the variables were found to be the stirrer speed n_{sr} =1300 rpm, initial product particle size x_p = 6 μm, and solid concentration of product particles C_v = 120 g/L. Ultra-fine grinding tests are reconducted in a RTSM-05 horizontal stirred mill using the process parameters optimized mentioned above. The results revealed that, after only t_g = 5 min of grinding, the product particle size P_{50} is 1.78 μm. Figure 7 shows the product particle size under different wet milling process parameters. Focusing on the wet milling efficiency, the preferred milling processes parameters at different concentrations, shown in Table 5, were screened for comparative analysis to simplify the amount of data for comparison. As can be seen in Figure 7, the optimized process parameters can achieve the minimum product particle size (P_{50} = 1.78 μm) and maximum solid concentration (C_v = 120 g/L) within the minimum grinding time (t_g = 5 min).

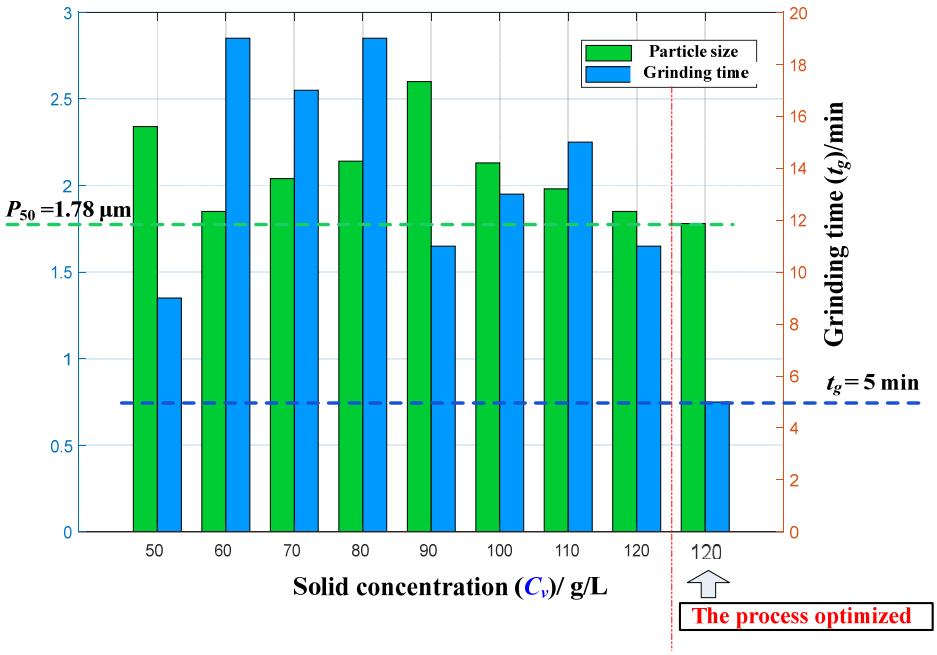


Figure 7. Comparative analysis under different process parameters.

4. Conclusions

The parameter optimization of WSMM to achieve ultrafine grinding with improved milling capability and quality is crucial. The objective of this paper was to develop a parameter optimization methodology for multiple-objective WSMM optimization. A mechanistic model was proposed, and the specific energy was quantified according to a stressing model. To improve the single-particle comminution rate, the specific stress intensity and specific stress number were proposed. The objective of parameter optimization is to achieve the maximum specific stress intensity and specific stress number under the constraint of the product particle size. The SFLA was applied to find the optimal parameters. The performance of the proposed strategy is validated using two case studies in different industrial optimization scenarios. The result of the first case study illustrates that, in comparison to a

quadratic programming-based response surface methodology, the proposed SFLA-based stressing model greatly enhances the wet grinding efficiency (decreasing P_{80} from 3.28 μm to 2.88 μm). In the second case study, the parameter optimization under different feed particle sizes and different productivities was discussed. The results confirmed that the optimized parameters can achieve the minimum particle size (P_{50} = 1.78 μm) and maximum solid concentration (C_v = 120 g/L) within the minimum grinding time (t_g = 5 min). Notably, the core value of this study extends beyond laboratory validation, providing critical guidance for industries utilizing stirred media mills. The RTSM-05 horizontal stirred mill used in Case 2 is an industrial-grade device, consistent with commonly used equipment in the industry, and its optimization results can directly serve as a parameter reference for similar industrial equipment. In industrial scenarios, determining processes for new materials typically requires repeated high-cost pilot tests; however, this model can quickly predict optimal processes using only laboratory-scale material property data and mill parameters, significantly reducing trial-and-error costs and shortening process development cycles. Meanwhile, the model's adaptability to different mill types (vertical and horizontal) provides a theoretical basis for enterprise mill selection—when enterprises need to switch between ore grinding (vertical mills) and chemical filler grinding (horizontal mills), the model can quickly match suitable parameters based on mill structure and material characteristics, avoiding the inefficiency of "one test per machine".

Furthermore, through collaboration with Anhui Root Intelligent Equipment Co., Ltd. (Suzhou City, Anhui Province, China), the optimization strategy proposed has initially demonstrated advantages in preliminary industrial applications: in the grinding production of materials such as HCC, it not only achieves precise control of product particle size but also improves production stability, providing a scalable theoretical framework for the optimal design and efficient operation of industrial-grade stirred media mills.

In summary, the "SFLA-stressing model" system constructed in this study effectively bridges the gap between academic modeling and industrial application. It provides a practical cost-effective tool for industries using stirred media mills to improve production efficiency and reduce energy consumption and costs, and it holds broad prospects for industrial application. The proposed approach is systematic and robust and can be integrated into WSMM architectures for parameter optimization in other complex WSMM.

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