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# Application of the product related stress model for product dispersity control in dry stirred media milling

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A B S T R A C T

Ground ultrafine and nano- materials are mainly produced by wet stirred media milling. The product related stress model can be used for the optimization of such grinding processes; the operational parameters can be related to the particle size or specific surface of the product. At the same time, for many materials dry grinding is required. During dry stirred media milling different problems arise, such as aggregation, agglomeration and particles sticking on the mill liners and on the grinding media. Consequently, the processes taking place in the mill and the effects of the operational parameters on the product properties are not as thoroughly known as in the case of wet stirred milling. In the present paper the application of the product related stress model for product dispersity control in dry batch stirred media milling is presented. Modifications of the models' main parameters such as stress intensity and stress number are introduced. The applications of the modified main parameters are presented for the optimization of the material and grinding media filling ratio, as well as functions for the characterization of the effect of the stress number and stress intensity on the product. With the introduced modifications, the product related stress model proved to be suitable for the characterization of dry batch stirred media milling.



## 1. Introduction

In the last decade demand has increased for fine (<50 μm), ultrafine (<5 μm) and nano (<500 nm) materials, especially in the pharmaceutical, food, chemical, paint and ceramic industries. The production of such ultrafine ground products, however, has some difficulties. The specific grinding work consumption increases exponentially with the grinding fineness. During ultrafine and nano grinding the extremely increasing specific surface is accompanied by the appearance of very high free surface energy, which leads to the aggregation and agglomeration of the particles. Sticking of the particles on the mill liners and on the grinding beads is also associated with this effect. The prevention of these milling efficiency impairing effects can be effectively carried out by utilizing wet grinding, but in many cases dry grinding is required. Thus, an effective and efficient solution to this dry grinding problem is still a target of research.

The properties of a bulk material can be divided into two main groups from the grinding point of view: (1) dispersity, characterized by the particle size distribution, particle shape, surface morphology and by the interfacial properties, and (2) structural properties, such as crystal structure, amorphous, microstructure, and impurities in the particle. The common purpose of grinding is to decrease the particle size or to increase the specific surface area; however, during grinding not only these properties can be changed, but other dispersity properties may be modified, including particle shape and the structural properties of the material. The above-described properties are collectively called "property function" (Rumpf, 1967), the control of which is known as product or particle engineering or design.

The control of the product properties made from dispersed materials can be done by the process function. A processing technology can be described by the process function (Peukert, 2004). When the comminution process is in the spotlight, two more functions can be defined - the mill and the material functions. The mill function is determined by the type, internal design and size of the mill and by the operation parameters. The material function can be characterized in two separate ways: on one hand there are the bulk properties of the material, like dispersity and structural properties; on the other hand there are the material properties of a single particle, like density, hardness, and Young's modulus. Because of high number of the parameters, for purposeful control the interrelated classification of the properties is necessary. (1) the type of stresses acting on the particles is determined by mill choice. The internal design and the operational parameters determine the stress intensity and the stress number; (2) the material properties of a single particle determine the breakage rate and breakage function (Peukert, 2004). The properties of the mill and the particle together determine the change in the bulk properties during grinding. The latter will determine the application properties of the product as well.

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The grinding kinetics based on the experience of dry batch ball milling (Juhász and Opoczky, 1990) can be seen in Fig. 1. Based on this approach, the particle size decreases significantly at the beginning of grinding, and the specific grinding work (or grinding time) is proportional to the produced specific surface, so to the ground materials' degree of dispersity. In this section, which is called the Rittinger section, the particle–particle interaction is very small. As the grinding progresses, with the increasing dispersity of the ground materials' the density of the location of the faults in the material decreases and the fracture hardness increases. Parallel to this, the sticking of the particles to the grinding media, to the mill liners and to each other begins, but the degree of dispersity increases further (section b in Fig. 1). After a long period of grinding, the increase in fineness is reduced to a minimum, and the decrease of the dispersity, so the coarsening of the ground material occurs (section c). The brittle materials are likely to mainly in this section undergo mechanochemical or crystal structure changes. Section b is called the section of aggregation, while c is the section of agglomeration. Aggregation can be defined as a weak attraction of the particles by van der Waals adhesion forces (mainly London-type dispersion forces) (Juhász and Opoczky, 2003). Agglomeration is an irreversible, solid attraction of the particles (by crystallization, welding and mechanochemical reactions), where chemical forces are exerted as well. The degree of aggregation and agglomeration can be reduced by the application of surface active grinding aids (Juhász and Opoczky, 2003).

The demand for ever-decreasing grinding fineness has resulted in the replacement of traditional ball mills with stirred media mills, which make possible the needed drastic increase in energy-transfer intensity along with the required decrease in the grinding media size. A stirred media mill is a high energy density mill which is used for industrial nano-size particle production, mainly in wet mode. The effectiveness of the mill has been established; however, its application in dry grinding is still limited today. The stirred media mill is an important milestone in the mill development, because while previously the movement of the grinding media was carried out by rotation (ball mill) or vibration (vibration mill) of the milling chamber, in this case the rotation of the concentrically or eccentrically placed rotor makes the grinding media move. As a result, the grinding media collide with each other; are pressed to the mill liners; and collide with the rotor. The particles of the feed material are fractured by the impact, pressure, collision and friction stresses. Changing the rotor velocity over a wide range – compared to the ball mill, where the stress intensity can be changed only in a limited range – allows the stress intensity to be changed over a wide range, which opens new horizons in the field of comminution.

German researchers (Becker et al., 2001; Blecher et al., 1995; Breitung-Faes and Kwade, 2013; Kwade, 2004, 1999a; Kwade and Schwedes, 2002; Kwade et al., 1996) created the so-called stress models for the description and explanation of wet stirred media milling. Based on their work, the grinding process can be described from two different points of view, based either on the particles of the feed or on the mill; according to the selected approach, mill-related and product-related stress models can be distinguished. The basic idea or principle of the product-related stress model is that for a given feed particle, the product quality and fineness achieved in a comminution or dispersing process is determined by how often each feed particle and its resulting fragments are stressed, and thus by the number of stress events of a feed particle, $SN_F$; and how high the specific energy or specific force at each stress event is, and thus by the stress intensity at each stress event, SI. The value of $SN_F$ and SI depends on the operation parameters. However, for the characterization of a mill, it makes more sense to define characteristic numbers, which are independent of the size and other properties of the product particles. The comminution behaviour of a mill is determined by the number of stress events which are supplied by the mill per unit time, the so-called frequency of stress events, $SF_M$; and the energy which can be supplied to the product particles by the mill at each stress event, the so-called stress energy, SE (Kwade, 2004).

Based on findings from wet stirred media milling (Kwade et al., 1996), the stress intensity shows the combined effect of three operating parameters (grinding media diameter, grinding media density, and rotor circumferential velocity), its value of SI is proportional to the kinetic energy of the grinding media. Fig. 2 shows that for a constant specific energy input the stress intensity SI determines the product fineness. Nearly no comminution progress is obtained at small stress intensities because the stress intensities are too low to break feed particles. It was proved in dry batch stirred media milling experiments that in this section rounding and shaping of the particles is carried out (Rácz, 2014a). However, multiple stressing and higher energy are required for grinding. With increasing stress intensity, the product fineness increases until a minimum is reached. At this fineness, the stress intensity has an optimum value. If the stress intensity is further increased while the specific energy input is kept constant, the product fineness decreases because of the number of stress events and, moreover, the energy utilization (newly generated surface related to energy input) of each stress event decreases. Since the specific energy can be considered as the product of stress intensity and number of stress events, the number of stress events decreases with increasing stress intensity at a constant specific energy. In this range of stress intensity, the improvement of the comminution due to the increase of stress intensity is less than the worsening of the comminution due to the decrease in stress number (Becker et al., 2001).

In another set of studies, a series of dry grinding tests were performed in a prototype continuous horizontal stirred mill to investigate the effect of operating parameters such as stirrer speed, feed rate, media filling and ball size, considering the degree of size reduction and the energy consumption. The test results showed that the stirrer

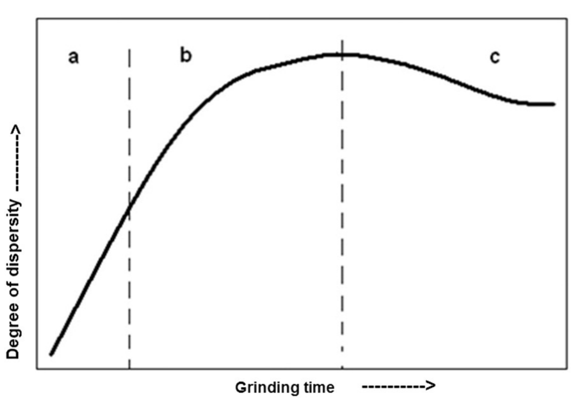


Fig. 1. Degree of dispersity as a function of grinding time (Juhász and Opoczky, 1990).

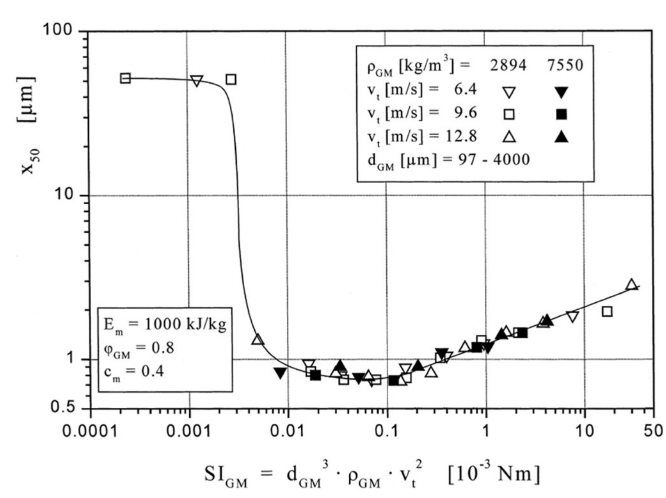


Fig. 2. Influence of stress intensity on product fineness at a specific energy input in wet grinding (Becker et al., 2001; Kwade et al., 1996).