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Solid state synthesis of nano-mineral particles

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ABSTRACT

Many researchers in academia and industries are interested in reducing particle sizes from few sub-micrometers to nano-meter levels. These nano-particles find application in several areas including ceramics, paints, cosmetics, microelectronics, sensors, textiles and biomedical, etc. This article reviews the present state of the art for solid state synthesis of mineral nano-particles by wet milling, including their operating variables such as ball size, solid mass fraction and suspension stability. This article concludes and recommends with a critical discussion of nano-particles synthesis and a few common strategies to overcome stability issues.

1. Introduction

Nano-particles are useful to unlimited applications in human life and it is drawing attention among researchers in academia and industries. Nano-particle has large specific surface area, high chemical reactivity and physical affinity as well as interesting optical, electrical and magnetic properties [1]. These nano-particles find application in several areas including catalysis, biomedical, biosensor, solar cell, ceramic, textile, electronics, water treatment and polymer nano-composite [2–19]. Nano-particles can be produced by the break-down (top-down) or the build-up (bottom-up) method, as shown in Fig. 1. All particle production techniques fall into three categories such as vapor-phase, liquid-phase and solid-state processes.

1.1. Vapor-phase synthesis of nano-particles

The vapor-phase synthesis of nano-particles involves the generation of vapor from the desired material and condensation of clusters and nano-particles from the vapor phase. The mechanism for particle formation is precursor vaporization, nucleation and growth stage and the schematic diagram is shown in Fig. 2.

The vapor may be generated by thermal, laser, electron beam, evaporation, etc. Different sources of energy can be used to decompose the precursor such as microwave plasma, laser pyrolysis, laser photolysis, combustion flame, etc. Vapor phase technique is used for producing nano-particles such as carbon black, silicon dioxide, titanium dioxide, which are used for tire and inks, additives in coffee creamers and polymers and UV-protecting gels and pigments for paints respectively. The size of the nano-particle is determined by the particle residence time, temperature of the vapor, precursor composition, and pressure. The smaller particle size is achieved by the following factors such as higher the catalytic activity (Pt support on Al_2O_3), higher the mechanical reinforcement (carbon black in rubber), higher the electrical conductivity of ceramics (CeO_2), lower the electrical conductivity of metals (Cu, Ni, Fe, Co, Cu alloys), initially increasing and later decreasing magnetic coercivity, finally superparamagnetic behaviour (Fe_2O_3), higher the hardness and strength of metals and alloys, higher the ductility, hardness and formability of ceramics; the lower the sintering and superplastic forming temperature of ceramics (TiO_2), higher the blue-shift of optical spectra of quantum dots (quantum confinement of Si) and higher the luminescence of semiconductors (Si, GaAs, ZnS:Mn^{2+}) [21]. The synthesis of nano-particles and their application of those nano-particles in carbon monoxide oxidation; magnetic property of FeAl and NiAl; nano-structured particulate films like catalysis, gas sensors; and electronics are studied [22–25].

1.2. Liquid-phase synthesis of nano-particles

The liquid phase synthesis of nano-particle is based on wet chemistry method and the schematic diagram is shown in Fig. 3. The particle formation mechanism is as same as in the vapor phase process. The liquid phase processes in nano-material production are precipitation, sol–gel process and hydrothermal process [26–28].

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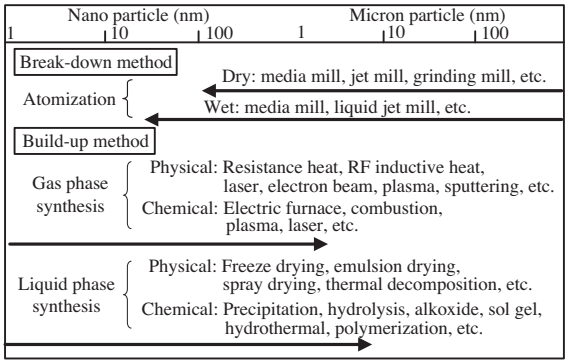

Fig. 1. Method of synthesis of nano and micron particle size [20].

The main advantage of liquid phase synthesis is it could be carried out at lower temperature than gas phase synthesis. The particle size, crystal shape and quality of product could not be controlled well by vapor process. But liquid process could be overcome these issues although, particles could aggregate during post calcinations step in sol–gel process.

Nano-particles such as βGa_2O_3, copper, gold, silver, metal oxide like lanthanum hydroxide, manganese oxide, $ZnFe_2O_4$, indium, iron oxide and palladium, SnO_2 are successfully synthesised by liquid phase technique [29–40]. The liquid phase enables to produce highly porous materials and mono-disperse product (uniform particle size) where as highly porous material could not be possible in gas phase reactors due to the high temperature. Moreover, the gas phase processes do not allow the synthesis of organic nano-particles. The gas and liquid phase methods can produce higher quality nano-particles but at a lower capacity in comparison to the solid phase (i.e., grinding method).

Gas phase and liquid phase synthesis of nano-particles were extensively reviewed [41,42]. Recently industries such as mineral, ceramic, pharmaceutical, paint are interested to produce bulk quantities of nano-particles. Hence solid state method is attractive for such industries.

1.3. Solid-state synthesis of nano-particles

Generally, grinding unit consumes more specific energy than other unit operation in the industries. However, the stirred ball mill consumes lower specific energy and desired nano-sized particle than ball mill. Fig. 4 shows the generic concepts of bottom-down nano-particle synthesis method. The production of submicron particle is studied in stirred ball/attritor mill [43–49]. Particularly, stirred ball mill is one of the major unit operations with minimum specific energy consumption in various industries such as minerals, ceramic materials, pigments, pharmaceutical, chemical products and microorganism [50–59].

2. Method of grinding

2.1. Dry grinding

Dry grinding with stirred ball mill has been far less common than wet grinding [60]. Nano-sized particles are obtained up to >200 nm by dry grinding method. Main disadvantages of dry grinding are the production of wider size range of particles and difficulty in installing classifier for separation of fine and coarse particles. Dry grinding methods have a few limitations in particle size reduction such as it (i) needs far higher investment costs, (ii) requires a classifier unit for separation of fine and coarse particle size, (iii) gives wider particle size distribution compared to wet grinding, (iv) specific energy consumption is higher than wet grinding method.

2.2. Wet grinding

Wet grinding is one of the top down approaches used for the production of mineral nano-particles. Wet grinding using stirred ball mill is advantageous for nano-particle production over other fine grinding techniques owing to ease of operation, simplicity of construction, high size reduction rate and relatively low energy consumption [61]. The stirred ball mill consumes 40–60% less energy than ball mill, because of the efficient use of grinding media where a force of 30–33 times bigger than the gravitational force is applied. However, most of the published works have used the average particle size as an indicator of particle size and not complete particle size distribution. There are few limited information is available for using average particle size such as (i) particle size distribution is based on following properties such as bulk density, dustiness and dispersion. But these properties are not estimated from average particle size. For example, average particle size (d_{50}) is same for two distributions, but particle size less than 10% (d_{10}) and particle size less than 90% (d_{90}) are different, (ii) it is difficult to get exact energy consumption for particle size distribution from conventional methods of size-energy relation because the size-energy relation is based on single particle size (d_{50} for Rittinger's law and d_{80} for Bond's law) and (iii) dispersion stability strongly depends on the particle size distribution rather than average particle size. Moreover, suspension/rheological properties are exactly obtained from particle size distribution rather than average particle size.

2.2.1. Effect of ball size

During comminution the grinding media size selection is of great importance. As grinding proceeds, the particle size decreases resulting in an increase in the number of particles in the system. However, the number of balls is the same in grinding vessel. Therefore, less number of particles is selected during each grinding event which reduces the specific rate of breakage. Earlier, an optimum ratio of ball size to particle size of 20:1 is studied and to provide the lowest median particle size (5 μm) [62]. The effect of ball size and other parameter such as ball density, solid mass fraction and pin tip velocity on production of micron particle size are studied using factorial design method [63–66]. It is inferred that desired micron particle size is obtained at decreasing ball size. However, small and large ball size is not effective for all the case. For example, there is no grinding progress when small size of grinding media are used (<0.838 mm); the stress intensity is too small to break a feed particle of limestone at each stress events for production of sub micron particle [67]. This was confirmed and studied of an

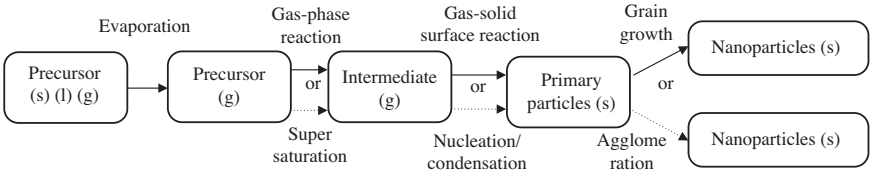

Fig. 2. Generic concepts of bottom-up nanoparticle synthesis process (vapor phase).