Review

# Introduction of Powder and Grain Handling Equipment and Its Applied Technologies

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# 1. Preface

The history of handling technologies of particulates (powder and grain), including crushing, mixing, kneading, drying, granulating, and classification, is lengthy, and the technologies have been utilized in various industrial fields as one of the important technologies to sustain modern industries. Seemingly simple particulate handling technologies are surprisingly profound and require a variety of know-how. In association with the increase of sophistication for such a particulate handling technology, newly developed equipment and/or application technologies have been required. Some pieces of particulate handling equipment that the author's company deals in are introduced in this article, along with their applied technologies.

# **2.** Classification of grinding machine and crusher (pulverizer)

Grinding and Crushing equipment (pulverizer) is roughly categorized into two types, dry-type and wet-type machines, each of which is characterized by its own advantages/ disadvantages. A dry-type machine is mainly applied to pulverize raw materials for refractory products. It is generally acknowledged that the limit of minimum particle size achievable by the dry-type crusher is several micrometers. Pulverizing to sub-micrometers of particle size is achievable by some innovative dry-type machines. When pulverizing to fine particles smaller than several-ten micrometers of particle size, however, difficulties of adhesion and/or aggregation of pulverized matters come to the surface. A dry-type machine, which is not good for pulverizing an object to a very fine particle size, has a big advantage of omitting a drying process. Since the drying process requires plenty of energy, dry-type crushing is desirous unless it includes a wet-type operation in the pre- and/or post-processes. On the other hand, a wet-type machine enables pulverization of much finer particulates with higher precision than a dry-type machine. In recent years, some wet-type machines that enable uniform pulverization/dispersion of particulate down to nanosize have shown up. They play an active role in the fields of electronic component material, electric battery material, pharmaceutical products, cosmetics, ink, fine ceramics, etc.

Therefore, depending on the characteristics of a pulverized raw material (size, hardness, mechanical strength, or abrasiveness) as well as on the required processing (particle size of pulverized particulate or particle size distribution), a different type of machine needs to be selected and several types of machines are combinedly utilized in multiple stages in some cases.

# 2.1 Dry-type grinding machines

The author's company sells dry-type grinding machines mainly for fine particle size powder products, which are roughly categorized into a media agitating mill and a media-less mill that does not use grinding media. The media agitating mill, in which grinding media such as balls (ball mill or attritor) are used, has been utilized for pulverization of refractory raw materials since a long time ago. Since large crushing energy can be added to materials to be crushed, the media agitating mill enables large quantities of pulverization of quite hard materials, such as ceramic or metallic oxides. However, abrasion of crushing members and contamination of powder produced from the abraded matters take place as confronting issues in pulverization by the media agitating mill. To avoid contaminating with iron components, ceramic balls or crushing members of other types of materials should be adopted for less abrasion. On the other hand, various pulverizing mechanisms, such as compression shearing (for a roll mill or a jaw crusher), an impact mill (for a hammer mill), a grinding mill (for a stone mill), autogenous grinding with mutual collision of crushed materials (for a jet mill or an air stream grinding mill), are applied for the media-less mill. Contamination with abraded matters can be minimized by the autogenous grinding method. Since the input crushing energy is small in the media-less crusher in comparison with the media agitating mill, production efficiency is inferior, especially for pulverization of fine powder products. Dry-type grinding machines that are supplied by the author's company are introduced below.

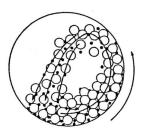
#### 1) Attritor

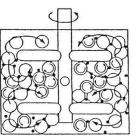
Attritor was developed by Dr. Szegvar et al. working for Union Process, Inc., USA, as a wet-type mill crusher in the 1940 s. The author's company (Mitsui Mike Machinery Company at that time) imported the technology and began the sales of Attritor in the 1960 s. In 1978, the technology for a drytype Attritor was imported from Union Process, Inc. Because of its structure and functions, it was acknowledged that the operability and safety of an Attritor were remarkably improved compared to those of a ball mill or a roll mill. Due to the effect

of centrifugal force compellingly applied to the agitating grinding media, much higher energy can be imputed than that in a ball mill or a vibration mill operation, resulting in a quite high comminution rate, which is almost ten times the comminution ability of a ball mill. Compared with a conventional ball mill, the structure of the Attritor is schematically shown in Fig. 1. Since a water cooling jacket was installed around the perimeter of a grinding tank, the exothermic heat induced by pulverization can be let out and the temperature of pulverized materials can be maintained at a certain level. Furthermore, utilizing the sealing structure of the Attritor grinding tank, pulverization under inert gas and a depressurized or vacuum atmosphere can be carried out. Thanks to the uniform pulverizing operation, various ingenuities are implemented in the shape and dimension of agitating arms so that pulverized matter is thoroughly and uniformly crunched with grinding balls without any retention in the agitating chamber.

#### 2) Alchemy

Alchemy is an independently developed grinding machine. In comparison with the Attritor whose agitating drive shaft is set in a vertical direction, namely, in parallel to gravity, the agitating drive shaft of Alchemy is, as shown in Fig. 2, set in a horizontal direction. Despite the assimilated structure to the Attritor, Alchemy was elaborately designed so as to agitate grinding media more uniformly. When a crushed material with high density, such as metal powder, is pulverized in the Attritor, the crushed powder tends to be unevenly distributed on the bottom of the grinding tank, resulting in uneven pulverization performance. In addition, due to the influence of grinding balls' own weight, uneven comminution energy distribution in a





Dependent on gravity

Centrifugal force effect by compelled agitating

Fig. 1 Structure of conventional ball mill (left) and Attritor (right).

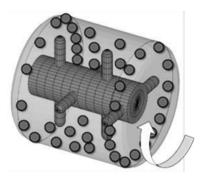


Fig. 2 Inner structure of Alchemy.

vertical direction takes place in the Attritor grinding tank. Such uneven distribution of powder and uneven griding energy cause a lowering of efficiency of comminution, which becomes more prominent in association with an increase in the volume of the grinding tank and is deemed as a scale-up risk. Contrarily, because of its horizontal-type configuration, problems in Attritor, such as uneven distribution of grinding media and powder product, were improved in Alchemy, which materializes roughly 3 times of comminution capability of Attritor and the scale-up risk reduced to less than half. Because of such advantages, Alchemy is thought to be a suitable grinding machine for the mechanochemical reaction or the mechanical alloying process, which are described later.

#### 3) Dynamic mill

The dynamic mill shown in Fig. 3 is a continuous dry-type grinding machine having over 30 years of supply record since its sales release. The Dynamic mill enables automated continuous comminution operation with 10-20 times of comminution capability of a batch-type ball mill. It also enables mass production of powder products with a minimum installation area. Unlike an air stream grinding mill or a jet mill, quite a small number of incidental facilities are required for the Dynamic mill. Specifically, only a raw material supply system is required. Due to this reason, its installation cost can be minimized along with the small installation area. At the same time, the maintenance time for equipment adjustment or cleaning is reduced. Since the internal structural components of the grinding tank of the Dynamic mill are compatible with ceramics, it is suitable for a crushing operation, in which contamination of powder products with iron components should be avoided. According to the precedent records, major applications of the Dynamic mill include grinding of inorganic substances, such as alumina, ferrite, silicon nitride, or silicon carbide. In recent years, many Dynamic mills have also been used in production facilities for powdered green tea (matcha). The amount of matcha production that had been achieved by grinding tea leaves with over hundred tea-grinding stone mills in the past can be conducted by only one Dynamic mill. Furthermore, powdered green tea with a similar level of flavor and/or feeling on the tongue to matcha ground by a tea-grinding stone mill can be produced by the ingenuities on milling operation conditions, which contributes to the recent

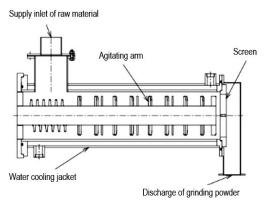


Fig. 3 Inner structure of Dynamic mill.

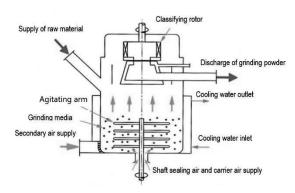


Fig. 4 Inner structure of Fine mill.

matcha boom.

# 4) Fine mill

Fine mill, in which the Attritor and a classifier are situated in lower and upper positions respectively, is a highly efficient continuous dry-type grinding machine. The structure of the Fine mill is schematically shown in Fig. 4. Powder products pulverized by Attritor in the lower position are transferred by carrier gas to the classifier imbedded in the upper position, where powder with a specified particle size is classified by a centrifugal rotor type classifier and then discharged and collected using a cyclone and a bag filter. Since the pulverized powder is rapidly discharged out of the crusher, the negative influence of fine powder on the grinding operation, such as adhesion or aggregation, can be suppressed, which contributes to the improvement of comminution efficiency. Due to the effect of the classifier, the particle size distribution of collected powder products is very sharp. Under such conditions, very fine powder with single micrometers or sub-micrometers of particle size can be steadily produced. The specified particle size can be easily changed and controlled by adjusting the rotation speed of a classifying rotor and the wind velocity of carrier gas. By accommodating internal components with contamination resistance materials, such as ceramic, pulverization that requires thorough prevention of metallic component contamination can be conducted. Similar to other dry-type pulverizers, an ancillary facility configuration of the Fine mill consists of a cyclone, a bag filter, and a blower. Unlike a jet mill, in which a large amount of compressed air is consumed, the Fine mill can be operated with small utility consumption. In this regard, the Fine mill can be rated as an energy-saving system with low comminution cost.

#### 5) Stream mill

Stream mill is an air flow type media-less pulverizer, in which, as schematically shown in Fig. 5, crushed matters are autogenously ground by their mutual collision, which is compelled by the powerful turning air flow generated by high-speed rotation of an impeller in the pulverizing vessel. Because of the minimized exothermic heat generation as well as the compactly designed simple structure and configuration, the Stream mill can be operated with lower running and energy costs than a jet mill in association with almost no contamination of powder products with other objects.

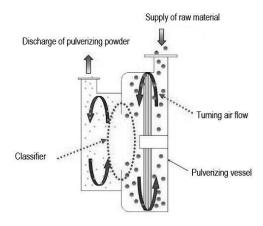


Fig. 5 Inner structure of Stream mill.

## 2.2 Wet-type grinding machine

Dry-type grinding machines are often applied to the comminution of refractory raw materials. While, as suggested previously, the wet-type grinding machine enables the pulverization/dispersion of much fine powder products with high precision. Recently, a large number of wet-type grinding machines that enable uniform pulverization/dispersion of particulate with nano-size particles have been supplied. Before introducing the wet-type grinding machines and crushers that are supplied by the author's company, a typical example of applied technology of the wet-type grinding machine is introduced. When a small amount of solid additive is used to coat the surface of the target base substance particles thoroughly and uniformly, the solid additive material that is solved in binder added solvent is firstly ground and dispersed to sub-micrometers or nano-size by the wet-type grinding machine. Afterwards, the resulting slurry including finely ground solid additive material powders is sprayed onto the base substance particles while agitating the particles in mixing flow in an FM mixer (high-speed fluidizing mixer), followed by drying under a depressurized atmosphere in the FM mixer to remove extra solvent components. Thus, target powder particles uniformly coated with the additive material are produced.

#### 1) Attritor

As suggested in the previous section on the dry-type Attritor, Attritor was initially developed as a wet-type crusher. By forceful agitating of media, Attritor exhibits almost ten times of comminution capability of a ball mill. In addition to the standard wet-type Attritor for a batch crushing operation, derivation equipment such as a continuous or circulation wet-type Attritor has been developed. Since relatively large size grinding media with roughly 10 mm of diameter is usable, raw materials with several mm of size can be efficiently pulverized to powder with micrometers of size. For 60 years from its sales release, demands for the wet-type Attritor have been continuing.

#### 2) SC mill

Using beads with 0.2-2 mm of diameter as grinding media, an independently developed SC mill enables the production of fine powder products with a micrometer or sub-micrometer of

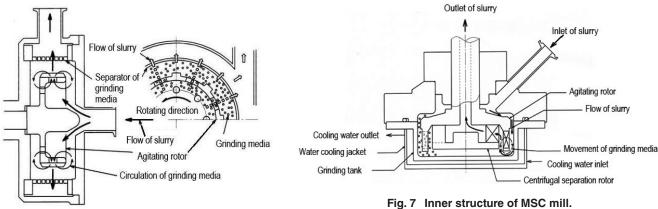


Fig. 6 Inner structure of SC mill.

size. Unlike a general bead mill that is designed to have a large L/D ratio (length/diameter of grinding vessel), the L/D ratio of the SC mill is 1/3. In association with the unique internal structure, the ideal movement of beads is accomplished in the grinding vessel of the SC mill, resulting in efficient pulverization/dispersion with low running costs. The internal structure of the SC mill is schematically shown in Fig. 6. The inner material of the grinding vessel accommodates ceramic, urethane, or resin parts associated with countermeasures for abrasion and/or contamination.

#### 3) MSC mill

Using beads with 0.015-0.2 mm of diameter as grinding media, the independently developed MSC mill enables the production of fine powder products with sub-micrometer or nano size. The structure of the MSC mill is schematically shown in Fig. 7. In the MSC mill whose internal structure was uniquely designed with a small L/D ratio, ground materials are steadily passed through an evenly agitated grinding media layer without a shortcut. Furthermore, small-sized grinding media and powder products can be surely classified by the independently developed centrifugal separation mechanism. The MSC mill is utilized for the production of electronic components that, in accordance with the expansion of smartphones or IoT devices, have been required further downsizing and high quality.

#### 3. Applied technologies by using a grinding machine

As applied technologies by using a grinding machine, mechanochemical effect, or mechanical doping that utilizes phenomena induced by grinding operation, mechanical alloying that utilizes collision/shearing energy induced by grinding media are cited. Even though these applied technologies are neither upto-date nor dealt with as sensational issues on center stage, their practical use is broadened in a variety of industries. In this regard, several examples of applied technologies using grinding machines are introduced in this section.

#### 3.1 Mechanochemical effect of grinding

In the early stage of the grinding operation, the specific surface of the ground material is increased in accordance with

the comminution. In the further grinding operation, the increase rate of the specific surface of ground material (comminution rate) is gradually lowered. When the specific surface of ground material reaches the maximum value (i.e. the comminution limit of ground material), the aggregation speed of fine particles exceeds the increase rate of specific surface, resulting in the progress of aggregation. In such a critical situation, input comminution energy is not used for the increase of the specific surface of ground material (i.e. size reduction) but is consumed in the aggregation of the fine particles. That is, the input comminution energy is consumed for cutting bonds between solid objects, resulting in destabilization (activation) of the solid objects, which causes aggregation of the solid objects with surrounding objects (fine particles) to a stabler condition of the whole system. In such a stage, in addition to the aggregation of fine solid particles, change of crystal structure in the solid particle accompanied by changes in its physical properties, absorption of gas on the solid particle, generation of radical (active species) on the solid particle surface, decomposition of the solid particle, exchange reaction or synthesizing reaction take place. Examples of the applied technologies in which such phenomena are utilized are briefly introduced.

#### 3.1.1 Unheated synthesis of cement-based material

Portland cement is produced by calcination of its raw material over 1000  $^{\circ}$ C. When the portland cement is produced without calcination or with calcination at a lower firing temperature, such a changeover is significant from a viewpoint of carbon neutrality, which is one of the attention-getting topics of recent days. As one of the possible changeovers, several research studies on the unheated synthesis of cement-based material with a mechanochemical effect are introduced.

# 1) Synthesis of tricalcium aluminate (C<sub>3</sub>A) by dry-type grinding <sup>1)</sup>

A mixture of three raw materials including raw lime (CaO), slaked lime (Ca(OH)<sub>2</sub>), and kaolinite (Al<sub>4</sub>Si<sub>4</sub>O<sub>10</sub>(OH)<sub>8</sub>) or gibbsite (Al(OH)<sub>3</sub>), which are properly weighed so as for Ca/Al molar ratio to be 3:1, is ground by stainless steel balls with 15 mm of diameter in a planetary ball mill for a specified time. Reactions induced by the grinding operation are expressed as follows:

$$0.5Al_{4}SiO_{10}(OH)_{8} + 3Ca(OH)_{2} + H_{2}O$$
  
-> 3CaO · Al\_{2}O\_{3} · 6H\_{2}O + 2SiO\_{2} (1)

$$2\mathrm{Al}(\mathrm{OH})_3 + 3\mathrm{Ca}(\mathrm{OH})_2 \rightarrow 3\mathrm{CaO} \cdot \mathrm{Al}_2\mathrm{O}_3 \cdot 6\mathrm{H}_2\mathrm{O}$$
(2)

The necessary grinding time for Equation (1), in which kaolinite is used, is shorter than that for chemical Equation (2), in which gibbsite is used. It is affirmed with the XRD pattern that  $C_3A$  hydrate is synthesized by roughly 30 minutes of reaction between kaolinite and slaked lime (Equation (1)). In the case of the reaction expressed by Equation (2), over 60 minutes of grinding is necessitated. In synthesis with gibbsite, 60 minutes of grinding materializes the hardest  $C_3A$  hydrate and the hardness of synthesized  $C_3A$  is gradually lowered as the grinding operation exceeds 60 minutes. The synthesized  $C_3A$ , which exhibits high activity after grinding, is hardened by adding water, accompanying the generation of overhydrate ( $C_3A(8-12)H_2O$ ). This behavior remarkably differs from that of calcined  $C_3A$ .

#### 2) Synthesis of calcium sulfoaluminate (CSA) hydrate<sup>2)</sup>

A mixture of three raw materials including slaked lime  $(Ca(OH)_2)$ , gypsum  $(CaSO_4 \cdot 2H_2O)$ , and gibbsite  $(Al(OH)_3)$ , which are properly weighed with 3:2:3 of  $Ca(OH)_2$ :Al $(OH)_3$ :  $CaSO_4 \cdot 2H_2O$  molar ratio so as to meet the chemical composition of ettringite  $(3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O)$ , is ground by stainless steel balls with 15 mm of diameter in a planetary ball mill. After 120 minutes of grinding, the synthesis of CSA is clarified with XRD analysis. In accordance with the grinding time, the Vickers hardness of the hydration hardened article is increased. The hydration hardened article of CSA synthesized by grinding for 6 hours exhibits similar hardness to that of commercially available CSA.

# 3) Synthesis of hydraulic powder from fly ash <sup>3)</sup>

Synthesis of hydraulic powder that, utilizing the mechanochemical effect, is fabricated by dry grinding of fly ash discharged from a coal fired power plant without calcinating process is investigated. That is, the fly ash admixed with 40 mass% of raw lime is ground by stainless steel balls with 15 mm of diameter in a planetary ball mill for a specified time. The hydration hardened article of powder product ground for 1 hour exhibits similar or higher hardness of cement clinker hardening article. The hardness of the hydration hardened article of powder product ground for 2 hours exceeds the hardness of the cement clinker hardening article.

# 3.1.2 Hydrogen production technology utilizing mechanochemical reaction

When an inorganic substance, such as silicon, iron, or aluminum, is ground in alkali-added water in a ball mill or a bead mill, in association with the pulverization of inorganic material, a large amount of hydrogen is generated by the reaction between the inorganic substance and water under an ordinary temperature and atmospheric pressure. Since the existing oxygen is mostly consumed by oxidation of inorganic substances in the reaction between inorganic matter and water, quite pure hydrogen gas is generated. Among many kinds of inorganic substances, silicon efficiently generates hydrogen gas and is easy to handle. It is expected that green hydrogen gas can be generated by a grinding machine driven by renewable electric power, created using sawdust of metallic silicon discharged and discarded from the manufacturing process of photovoltaic power generation panels or semiconductors, as well as a disposed solar panel whose service life runs out. It is inferred that the efficient grinding machine is an important key for such a project. Since an SC mill with a small L/D ratio exhibits eminent comminution efficiency, its contribution to such a project is expected.

# **3.1.3** Doping of nonmetallic elements in oxide (mechanical doping)

It is reported that a new function is elicited by doping an impurity element in the crystal lattice of functional oxide or by substituting the oxygen site in functional oxide with other types of atoms. For example, the band gap of zinc oxide, which is well known as a photocatalyst, can be lessened by substituting the oxygen site in zinc oxide with a nitrogen atom. With such a substitution of the atom, photocatalysis of zinc oxide, which is ordinarily activated by ultraviolet light, is responded in a visible light range. That is, such zinc oxide exhibits photocatalysis as a visible light-responsive type photocatalyst. Such doping treatment is conducted by the Attritor made by the author's company. With crushing the mixture of zinc oxide and urea (nitrogen source) in the Attritor for 1 hour, visible light responsive type photocatalyst powder is produced. To evaluate the possibility of mechanical doping by the continuous grinding operation, an experiment is conducted with a Dynamic mill. The light responsive type of zinc oxide photocatalyst with similar quality is successfully manufactured in the continuous grinding operation, which indicates a high possibility of industrialized mechanical doping.

#### 4. Classification of mixer and kneader

A variety of mixing or kneading machines are roughly categorized into two types. In one type, the mixed/kneaded material is agitated by an impeller or rollers in the fixed vessel. In another type, the mixing/kneading vessel itself is rotated. There exists a mixing/kneading machine in which the above two types are combined. That is, the mixed/kneaded material is stirred by the impeller or rollers in the rotating vessel.

The mixing operation is classified into dry mixing and wet mixing. Since dry mixing is usually applied to the mixing of refractory raw materials, the introduction of wet mixing is omitted in this article. For mixing and kneading operations, a single-purpose machine is applied in many cases. For some refractory application uses, however, mixing several kinds of raw materials and kneading with a binder added are simultaneously carried out in one machine. In this regard, the mixing/kneading machines which are supplied by the author's company are introduced.

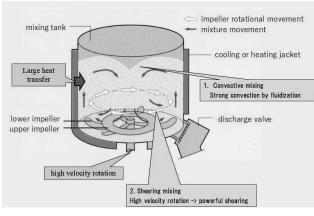


Fig. 8 Structure of FM mixer.

# 4.1 Mixer/Kneader

## 1) FM mixer

Since 1962 when the FM mixer technology was imported from Henschel company, Germany, the author's company has been supplying over 12,000 FM mixer which represent mixing/ kneading machines. Among its customers, the FM mixer is familiar as a Henschel mixer. The FM mixer features a largecapacity motor and a robust shaft design, which enables precise mixing/kneading in a short time by high-speed fluidized agitation. For accommodation to various mixing/kneading operations, several dozen kinds of impellers are prepared, for instance, to give powerful shearing force to the mixed materials by high torque agitating. As shown in Fig. 8, the jacket structure is installed around the perimeter of the mixing tank so as to enable heating or cooling during operation. Since vacuum, depressurized, or inert gas atmosphere corresponding to the tank, addition of chopping and/or scraping functions, anti-abrasion specification or 250  $^\circ\!\!\mathrm{C}$  of high-temperature proof specification are selectable as an option, not only mixing/kneading operation but also over ten kinds of processing, such as drying, granulating, coating, surface modification, conjugation, or pulverization can be performed by one multi-function FM mixer. The maximum mixing/kneading tank capacity of up to 8,000 L has been sold.

# 2) Conpix

Conpix is a continuous mixing machine with similar performance achieved by the FM mixer. Due to the independently developed design of a Conpix mixing tank, continuous and precise mixing/dispersion is achieved by adequately controlling the residence time of mixed materials in the chamber while minimizing the shortcut. Compared to an FM mixer with a similar level of mixing/kneading capacity, Conpix can be installed in a narrow space less than half of the FM mixer installation space, in association with a low-powered electric motor whose capacity is less than the motor used in the FM mixer. In this regard, Conpix is rated as an energy-saving and space-saving machine. The structure of Conpix is schematically shown in Fig. 9.

### 3) Kneadex

As shown in Fig. 10, in which the kneading flow in Kneadex

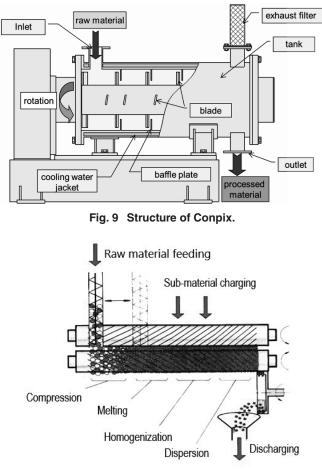


Fig. 10 Kneading flow in Kneadex.

is schematically illustrated, Kneadex is an open-type continuous kneader with a pair of kneading rolls. Despite it has no mixing function of powder materials, Kneadex is characterized by unique kneading functions. Because of the intentionally set differences in temperature and rotation speed between the two rolls, supplied raw materials are steadily entangled at one of the rolls into a small gap between the two rolls, and transferred to a discharge port associated with the spiral groove carved on the surfaces of pair of rolls while kneading. Due to powerful compression and shearing action induced by the small gap between the two rolls and the spiral groove, uniform kneading of the mixture with a high filler blending ratio can be carried out smoothly. Since kneading of the mixture is not done at a bank in an ordinary kneading machine with open rolls, turn-around operation is necessitated and charging/discharging operations should be repeated several times to knead, which requires skills and experiences associated with concern for safety. Because of the effect of the spiral groove carved on the surfaces of the rolls in Kneadex, kneading action at the bank is induced. As a result, not only ordinary mixtures but special mixtures, whose kneading is not performed by an ordinary extruder because of the large difference in viscosity between the raw material and the filler, can be uniformly kneaded. It is noted that Kneadex can operate continuously as well as automatedly with secured safety. For reference, kneading examples by Kneadex are listed as follows:

• Kneading of PP resin, in which talc is mixed with 85 mass% of high blending ratio, followed by pelletizing of the kneaded

product;

- Kneading of phenol resin, in which fine silica or alumina powder is mixed with 70-90 vol% of high blending ratio;
- Kneading of the mixture, in which 15 mass% binder such as wax is admixed to alumina-zirconia powder. In comparison with the kneading operation in a conventional kneader, the viscosity of the kneaded product is lowered to less than half, in association with better moldability which enables injection molding; and
- Kneading of fibrous filler, such as cellulose nano fiber, glass fiber or carbon fiber, in which the filler is uniformly kneaded into resin while untangling the bundles of fibers.

#### 5. Applied technologies utilizing mixer/kneader

In addition to mixing/kneading, various functions can be implemented in the FM mixer that is introduced in the previous section. Touching its derivation equipment, we describe the typical examples of applied technologies of the FM mixer.

#### 5.1 Drying treatment

Because of the jacket structure harnessed onto the FM mixer in which heating media, such as steam, hot water, or oil, can circulate for mixing/kneading operation in the heated-up tank, drying of slurry and/or removal of excess water or solvent components in the powder can be achieved. Since the mixing tank can be used in a vacuum or depressurized atmosphere, the kneaded mixture can be efficiently dried. As schematically illustrated in Fig. 11, the evaporated solvent is condensed by the condenser to be collected. Because of such a system configuration, drying treatment can be carried out safely without leaking the organic solvent out of the system.

Figure 12 shows changes in the powder (slurry) temperature, the moisture content in the mixture, and the power requirements in drying treatment of 100 kg of ceramic slurry with 50 % moisture content under a depressurized atmosphere in the FM150 type mixer with 150 L of inner tank volume. Because the boiling point temperature of water is lowered under a depressurized atmosphere, drying treatment is carried out at roughly 50  $^{\circ}$ C, and the moisture content in the mixture is lowered to 1 % or lower by 90 minutes of operation. Since the lower the moisture content is, the higher the viscosity of the mixture is, the ceramic slurry is coagulated to a pasty condition like a rice cake or a bread dough and, finally, to a coagulated lump with a dumpling shape. At around 60 minutes of operation time in Fig. 12 when the slurry is coagulated to the dumpling shape, power requirement is rapidly increased. Since quite large power is required in such an operation stage, the mixing of a dumpling shape mixture is often suspended because of overload suspension by a deficiency in torque or in the strength of the impeller or the shaft in an ordinary mixing machine, and as a result, the drying process cannot be completed. On the other hand, due to the powerful torque and the rigidly assembled structure, such difficulty is overcome and the dumpling shape of the mixture is altered to large, granulated particles in accordance with the progress of drying in the FM mixer. Since it is not easy

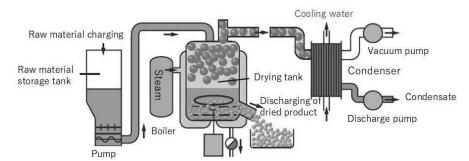


Fig. 11 Drying under vacuum or depressurized atmosphere in FM mixer.

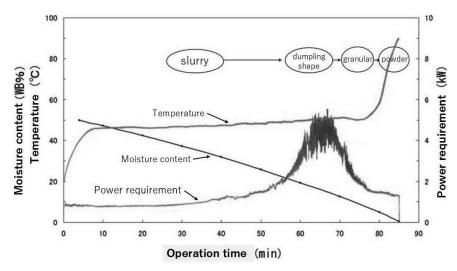


Fig. 12 Drying of ceramic slurry with FM mixer.

for moisture in the granulated particles to diffuse from the particle surface and vaporize, the drying rate tends to decrease at this stage. However, because of the cracking of granulated particles into powder by strong shearing force, drying is carried out quickly in the FM mixer. Furthermore, the increased fluidity of mixtures by cracking into powder induces a sharp drop in power requirement for mixing, and a rapid temperature rise when moisture evaporation ceases along with the completion of drying. By feeding back monitored values of the power requirement of the FM mixer and the temperature of the mixture to the drying control system, an automated operation can be accomplished. Unlike hot-air drying, for instance, by a spray dryer, the heat loss of exhaust gas is minimized in the drying treatment by the FM mixer. When drying is conducted in a vacuum or depressurized atmosphere corresponding to the FM mixer tank, the heat dissipation is further reduced because of the drying at a low temperature. It is noted that, despite the tank atmosphere, drying in the FM mixer is done efficiently with a constant drying rate because of the high thermal transmittance ratio induced by the powerful convection as well as the steady cracking of coagulated lump into powder.

The drying treatment in the FM mixer is accomplished by heat exchange from the heating medium circulating in the jacket installed around the perimeter of the mixing tank and the exothermic heat induced by agitating. In the drying treatment under a vacuum or depressurized atmosphere, heat transfer by air in the mixing tank is reduced or lost. In addition, the floating behavior of the powdery mixture is worsened under a depressurized atmosphere. Due to such reasons, overall heat exchange efficiency from the jacket to the mixture is reduced. As a countermeasure for maintaining efficient drying in the FM mixer, micro-wave is radiated to the mixture in the tank for direct heating. In comparison with the drying treatment by heating media circulating in the jacket, the drying rate by microwave radiation in the FM mixer is improved by almost double. When the drying by heating media and the drying by microwave radiation are combined, the drying rate is improved by four (4) times. Such powerful drying can be applied to the drying of adsorption water or dehydration of hydrate, both of which usually require huge energy.

#### 5.2 Coating, surface modification, and composite process

In the kneading treatment, the wettability of the particle surface needs to be chemically bettered by means of the addition of a third material such as a surface active agent, coating of the particle surface, or surface modification. Four (4) methods for particle surface coating and surface modification using the FM mixer are shown in Fig. 13.

In method (1), a liquid surface active agent or a surface modifier is sprayed or dropped bit by bit on particles that are mixed in a flow in the FM mixer to thoroughly and uniformly coat the particle surface, with care so as not to progress to granulation or coagulation. Afterward, the particles are dried in the FM mixer to evaporate the solvent matter so as to stabilize the coating of the modifying agent. A special coating like a

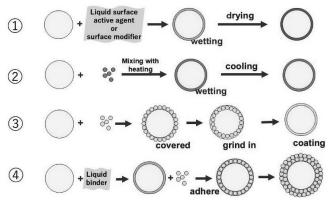


Fig. 13 Coating process of particles with FM mixer.

silane coupling treatment, in which a surface modifier is hydrolyzed by heating, is enabled in the FM mixer utilizing its functions for agitating at a constant temperature, cooling and drying after treatments, and retrieval and collection of the condensed solvent.

In method (2), a coating material that exists as a solid matter at room temperature and melts at an elevated temperature, such as wax or resin, is used to coat the particle surface. The mixture of target particles and a solid coating agent is agitated for mixing in the FM mixer while heating up to make the uniform coating on the particle surface with the melted coating agent. Unless adequate agitating conditions or an impeller is selected for the coating treatment in the FM mixer, granulation, adhesion or segregation of the coating agent is unintentionally caused. After completion of the coating, the coating agent is solidified by cooling the tank.

In method (3), a fine solid coating agent is shot onto the surface of target particles for coating. In the coating process, the solid coating agent that is shot onto the particle surfaces with quite high speed is rubbed onto the particle surfaces with strong shearing and impact force materialized by  $40 \text{ m} \cdot \text{s}^{-1}$  of tip speed in the standard type impeller to be immobilized. Furthermore, under the quite powerful shearing materialized by  $80-100 \text{ m} \cdot \text{s}^{-1}$  of ultra-high tip speed in a special type, conjugation applied coating can be more steadily conducted. A specialized mixer, "Composi," is developed for such a conjugation treatment. Granulation with no use of a binder is enabled by "Composi."

In method (4), a fine solid coating agent is used to coat target particle surfaces with a liquid binder. Application of the method (3) is restricted to coating for particles of relatively soft materials that exhibit some plasticity, such as resin, organic substances, or metallic materials with low hardness. While, method (4) is applicable to coating for particles of quite hard materials, such as ceramic, mineral substance, or metallic oxides. Firstly, target particle surfaces are uniformly coated with a liquid binder such as in method (1). After that, a fine coating agent is supplied under the fluidized mixing condition and adhered to the target particle surfaces coated with the liquid binder. By repeating such coating processes, the coating layer thickness can be controlled arbitrarily.

# 6. Conclusion

Over a half century from their sales start, Attritor and FM mixer have been playing an active role in a variety of industry fields. Based on fundamental technologies, the brush-up of equipment has been continued in association with application development so as to respond to requirements in the market, resulting in the development of derivation equipment, such as Alchemy or Composi. The handling technology of powder granules is surprisingly profound and there are many other types of equipment or application uses, which are not described in this article. We would like to improve informative communication

like this opportunity and progress our ceaseless development work so as to contribute to the evolution of the refractory industry.

### Reference

- 1) J. M. Filio, R. V. Perucho, F. Saito, M. Hanada and Y. Ito : *Materials Sci. Forum*, Vols.225-227 (1996) pp.503-508.
- 2) G. Misaka, F. Saito, M. Hamada and H. Ito : *Inorganic Materials*, 3 115-120 (1996).
- 3) K. Yone, F. Saito, Y. Waseda and T. Narita : *Journal of the Society of Powder Technology, Japan*, 35 [9] 639-645 (1998).