The Angmill Mechanofusion System and its Applications

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1. Introduction

In recent years, research on and the development of new-generation industrial materials have been done actively in various kinds of industries, including fine ceramics. It is well-known that most of the raw materials of new-generation materials are handled in a powdery form because the particulate state of the solid is the most suitable one for the development of new-generation materials.

The most characteristic aspect of the powder is the size of the component particles. Today, a growing interest is being taken in ultrafine particles on the order of an ångstrom, as well as in submicron size particles.

On the other hand, the creation of new-generation materials by combining different powder materials has been of great importance as indicated by the successful development of various composite materials and functional devices.

The authors, who have been studying this subject for many years, developed an ultrafine "Angmill" grinding system making the best use of the most effective mechanism for fine grinding (1983) 1). Furthermore, the authors discovered that the Angmill system is also useful in powder treatment techniques. They introduced some of its applications in August 1986 2).

Lately, they have succeeded in developing a "Mechanofusion system" resulting from research on the application of the high-powered mechanical energy of the Angmill system to powder treatment.

"Mechanofusion" treatment is defined as the technique of creating particulate materials with new physical properties by mechanochemical surface fusion as a result of the strong mechanical force acting on the surface of different kinds of particles. Some typical examples from the numerous possible combinations of the materials handled by this system and the consequent change in powder properties will be discussed in this report.

2. The Angmill principle

The Angmill system was originally developed as an ultrafine grinding system with a simple structure based on attrition under strong compression. Prior to discussing the mechanofusion effect, its grinding mechanism will be briefly explained.

The main grinding chamber of the Angmill system rotates at a high speed. At the center of the mill, the inner pieces are fixed with a certain clearance against the inside wall of the chamber. The head of the inner pieces have

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Fig. 1 The principle of the Angmill
a smaller radius of curvature than that of the chamber. The material compacted by centrifugal force onto the inside wall of the chamber is compressed into the clearance and receives complicated forces in various states, such as compression, attrition, shearing and rolling (Fig. 1).

Figure 2 shows the variation in the tangential force acting on the tip of the inner piece with the loading weight at various rotation speeds. The result was obtained using a smaller type of mill having a chamber diameter of 350 mm and a talc powder with an average size of 44 microns as the feed to be ground. As seen from the figure, the point of increase of the tangential force on the inner piece depends upon the rotation speed.

Figure 3 shows the bulk density of the talc powder in the rotating chamber, which was calculated from the loading weight at the high point in Fig. 2 and the annular volume between the inner piece tip and the inside wall of the rotating chamber. It is understood that at 800 r.p.m. the talc powder has a higher bulk density than the packed bulk density obtained by a tapping of 1.4 g/cm³. The ground product has an average size of 0.5 μm.

On the other hand, the loading weight at the high point did not depend upon the rotation speed in polystyrene beads having an average size of about 450 μm. Besides, the apparent volume of the material was almost the same as the above-mentioned annular clearance volume in the rotating chamber.
Figure 5 Variation with rotation speed \( (n) \) in the radial and tangential forces acting on the inner piece tip

Figure 4 shows the relationship between the rotation speed and the tangential force acting on the inner piece at the maximum charge of the material. The polystyrene beads were not ground at all under these conditions, though they are rather brittle among the plastic resins. In this case, the large amount of energy consumed was not utilized for size reduction but was converted into heat. The heat produced by the intense attrition between the particles is supposed to be closely related with the mechanofusion effect, discussed in the next section.

In Fig. 4 is also shown the variation in the tangential force on the inner piece with the rotation speed at a certain charge of talc powder which was obtained from Fig. 2. The tangential force of talc at the same revolution speed was three times greater than that of polystyrene beads. This fact seems to be attributed to the difference in the physical properties of the feed material and would help to elucidate the mechanisms of fine grinding as well as of the mechanofusion effect.

Figure 5 shows the variation in the radial force acting on the inner piece as well as the tangential force measured independently at a charge of 550 g of polystyrene beads at the rotation speed using the same type of machine as the foregoing. The log-log plot of the radial force against the rotation speed shows a linear relationship, but that of the tangential force tends to be bent. The radial force is related to the compressive force working on each particle, while the tangential force is related to the shearing force or attrition.

3. Examples of mechanofusion effects

Mechanofusion treatment is a technique to create new powder materials by the mechano-chemical surface fusion of different particles. The authors have already investigated combinations of more than 100 kinds of materials, but there are still countless possible raw materials yet to study. Several examples of mechanofusion will be reported later, though some of the material properties, the mixing ratio or the applications cannot yet be published in detail because most of them are new materials still under development. (The size after a material name indicates the average value.)

3.1 Polymethylmethacrylate (PMMA) \((5 \mu m)\)
\[+\text{TiO}_2 \ (0.015 \mu m)\]

By the treatment of the mixture of these two materials with the Angmill system, the finer TiO\(_2\) particles are firmly fixed onto the surface of the PMMA spheres and do not separate even by violent agitation in the water.
This combination was the first object of our research on mechanofusion and brought to light interesting changes in various physical properties.

Photo 1 shows the SEM picture of two kinds of raw materials. The surface of the particles produced by mechanofusion treatment is shown in Photo 2. Photos 3 (a) and (b) are TEM pictures of the treated particles sliced with a microtome.
The mapping of the chemical elements with an X-ray microanalyzer (Photo 4) indicates that the element Ti is scattered uniformly on the surface of the PMMA particle.

The PMMA powder treated with TiO₂ by mechanofusion flows like a fluid, and the angle of repose becomes nearly zero (Photo 5), though both raw materials have very low flow-ability (Photo 6).

Figure 6 shows how the angle of repose of the product measured by an injection method with a Powder Tester changes as the content of TiO₂ increases. In the figure, the result of the product of mechanofusion treatment is compared with one that had undergone a several-hour coating operation using a pot mill. The angle of repose of the mechanofusion product decreased to nearly 0 degrees at a TiO₂ content of 10%, while that of the mixture processed with a pot mill did not change so much.
Figure 7 shows the variation in the rate of penetration into the powders obtained by mechanofusion. The penetration rate was measured with a Penetanalyzer, which has been developed to evaluate the wettability of powders by measuring the penetration rate of a liquid into a powder bed. No water is sucked up into the hydrophobic PMMA powder. The results show that the wettability is improved by increasing the TiO$_2$ content. The PMMA powder mixed with 30% TiO$_2$ using a pot mill indicated a one-fifth penetration rate of the product by mechanofusion treatment containing 10% TiO$_2$.

### 3.2 Polystyrene resin (PS) + carbon black

A polystyrene resin was prepared to have an average size of about 10 $\mu$m by grinding and classification. The temperature control is the most important point in the mechanofusion operation. The ground particles having an irregular shape are rounded to improve their flowability by mechanofusion, as seen in the electron micrographs (Photo 7). This combination could be applied to the development of materials for electrostatic copying machines.

### 3.3 Ground PS (10 $\mu$m) + PMMA (0.5 $\mu$m)

Photo 8 shows the progress of the mechanofusion process with the lapse of time, where the PMMA spheres having an average size of 0.5 $\mu$m are being fixed onto the surface of the ground polystyrene particles used as a raw material in the above 3.2. It is observed that the PMMA particles having a higher melting point are buried in the polystyrene spheres having a lower melting point.

### 3.4 SiO$_2$ (1 $\mu$m) + TiO$_2$ (0.015 $\mu$m)

The cross-sectional electron micrograph of the particle obtained by the mechanofusion effect (Photo 9) displays the TiO$_2$ particles firmly fixed onto the surface of the SiO$_2$ sphere. The material treated by mechanofusion was not separated by agitation in water but settled out to leave clear water, while the mixture processed by a pot mill produced a turbid suspension containing fine TiO$_2$ particles separated from the SiO$_2$ spheres.
3. 5 PMMA (5 μm) + PTFE (0.1 μm)

The polytetrafluoroethylene (PTFE) material used for mechanofusion was a product ground by a jet mill and had an average size of about 10 μm. The magnified picture proved that the particles consist of firmly agglomerated spheres with an average diameter of about 0.1 μm (Photo 10a). When the PTFE agglomerates were processed by the Angmill system with the PMMA powder in a certain ratio, they were dispersed to the single spheres and fixed onto the surface of PMMA particles by mechanofusion (Photo 10b).

Figure 8 shows the charge distributions of the raw material of PMMA and the treated product measured with an E-SPART analyzer (Particle Charge Spectroanalyzer). The PMMA particles were almost uncharged, while the PMMA treated with the PTFE spheres showed a strong negative polarity.
3.6 Mechanofusion of three kinds of powders

Photo 11 shows a polymer particle with an average size of about 10 μm covered with carbon black by mechanofusion. The treated polymer powder was further processed with PMMA particles (0.5 μm) to produce a three-layer structure (Photo 12).
3. 7 Strong dispersion effect (Precision mixing)

As described in the foregoing section, the Angmill system achieved the complete dispersion of the agglomerates by inducing a powerful shearing force even on the submicron particles.

This mechanism can be applied to the precision mixing of fine powders, such as pigments and dyestuffs for paint materials.

![Photo 13 upper: Ground polymer lower: Treated product (about 15μm)]

3. 8 Particle shape control (Sphericalization)

One of the remarkable features of this system is the rounding effect of the irregular particles, as shown in the foregoing polystyrene resin case. Photo 13 shows the raw material of a polymer obtained by grinding and the treated product. The latter consisted of roundish particles of nearly the same size. The fine fragments in the raw material seemed to be fused and combined with the coarser core particles.

4. Mechanofusion production system

Figure 9 shows the flowsheet of the mechanofusion system. The raw materials are fed after weighing and mixing in a certain ratio into the treating machine using a modified Angmill system. In the machine, treatment is done in a batch operation for a certain time. Then the product is taken away swiftly from the main chamber and collected for delivery.

The entire system is controlled automatically with a computer.

5. Conclusions

The Angmill system has various functions, such as ultrafine grinding, mechanofusion, intense dispersion, sphericalization and so forth. These seem to be attributed to the strong compression and attrition forces, the accompanying heat as well as the electrostatic effect working in a complicated way on the individual fine particles in the range of several microns.

It is especially of great interest to be able to create new materials through mechanofusion as a consequence of the mechanochemical action on various powders having different physical properties.

A great deal of energy is consumed in the research and development of new-generation materials in both the academic and industrial fields. The mechanofusion technique is expected to be increasingly applied to the creation of new powder materials in the future.

References

Fig. 9 The flowsheet of the Angmill Mechanofusion System