Improvement of The Flowability of Particles and Development of Conductive Material by Mechano-Fusion Method

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Keijiro TERASHITA*, Kiminori UMEDA** and Kei MIYANAMI*

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An experiment applying different time frames and different rotating speeds for a casing were conducted to clarify the mechaism improved liquidity of composite particles produced by mechano-fusion. A method to produce conductive particles by dry coating was investigated. As a result, it was found that composite particles with superior flowabilty are obtained from source particles appropriately sprayed with fluidizer on the surface, while weak mechanical energy and excessive energy, respectively, cause insufficient spray of the fluidizer and burying of the fluidizer in the source particles so that adequate products are not obtained. Possibilities of conductive for semiconductors and applications made by the dry coating of conductive fine powder on resin particles were indicated.

1. Introduction

Surface treatment to give new uses and higher efficiency to powder particles is attracting wide attention while the development of functional materials made of composite particles have been reported in a number of research papers[1]. One approach to surface treatment of particles and making composite particles is the mechano-chemical process[2~4]. This process involves one apparatus for mechano-fusion[5,6], which improves flowability of the powder by compounding two different kinds of power (PMMA and titanium oxide). the mechanism of this process, however, has not been clarified.

Electromagnetic interference (EMI) has been controlled since December 1988 in Japan. The giving of conductivity to plastic insulators used in the electronic equipment housings is to prevent EMI[7~9], which has been a serious issue.

The writer has obtained successful results in this respect by kneading conductive fillers in resin. A dry coating conductive fine powder on resin particles by mechano-fusing will simplify the method. In this experiment, the angle of repose and outflow speed of flowability of the composite particles produced under different treatment times and rotating speeds were investigated while electrification, which seriously affects powder flowability, was investigated by an E-spart analyzer. The results of

* Department of Chemical Engineering, College of Engineering

** Mita industrial Co., Ltd.

the experiment help to analyze the mechanism of flowability. The results of the experiment on dry coating conductive fine powder on resin particles by mechano-fusion are reported below.

2. Apparatus, method and powder sample

The mechano-fusion process (Ang Mill) was adopted (Figure 1). This apparatus primarily used a compression/frictional crushing and grinding machine. The inner piece of smaller radii of curvature than the apparatus casing are fixed on the casing's inner wall at certain distances. The object materials are pressed against the inner wall of the casing by centrifugal forces caused by the high-speed rotation and further by strong mechanical energy (shearing, compression, rolling) which brings together compound and dry coat particles.

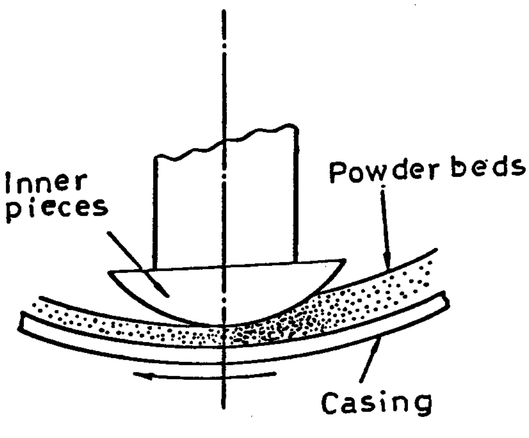


Fig. 1 Schematic diagram of the experimental apparatus

Various rotation speeds of the casing and different treatment times were applied to the powder sample (not premixed) in the casing to prepare mixed particles by a high-speed mixer (16.7 rps, 120sec.) and to compare the flowability with the composite particles produced by mechano-fusion.

The power characteristics tester and the E-spart analyzer were used to assess the flowability of particles made by mechano-fusion and the mixing process, respectively.

The conditions of the experiment for the composite particles made by the mechano-fusion process are shown in Table 1, while the powder samples are shown in Table 2. Corn starch is the source particle while silica particles are the fluidizer. The dry coating experiment conditions and powder samples are described below.

Table 1 Experiment conditions

corn starch	50.0 g
silica	0.5 g

Table 2 Powdem samples

corn starch	$D_{50}=6.9\,\mu m$
siqica aerogile	$D_{50}=2.0\,\mu m$

3. Adjusting and assessing the flowability of powder particles

Various rotation speeds for the casing were applied to make composite particles. The relation between the angle of repose ϕ_r and the rotation speed N are shown in Figure 2. The relation between outflow speed F and N is shown in Figure 3. The treatment time t was 300 seconds. The dashed line shows the physical properties of mixed particles produced by high-speed mixing. Figure 2 indicated that N=16.7 rps approximately and yields composite particles with an optimum flowability showing a small angle of repose while at N=33.3 rps the angle of repose is radically large, makig the flow inadequate, implying that the silica particles are buried in the source corn starch by strong mechanical energy (sharing, compression) so that it no longer plays the role of fluidizer (roller, slide). N=5.0 rps provides weak shearing force and compression impact so that the fluidizer is not sufficiently dispersed in the source particle, making flow inadequate. The condition of dispersion of silica particles (fluidizer) was measured by scanning electronic microscope. It was then understood that composite particles with optimum flowability are obtained when enough mechanical energy is available to disperse fluidizer sillica particles on one hand, while excessive energy is not charged to bury the sillica into the source particles on other hand. These conditions are found at a rotary speed of N=16.7 rps and 25.0rps. The flowability of the composite particles was found to be superior to mixed particles obtained by the mixer, implying that Ang Mill is a mixer which disperses fine powder in optimum uniformity.

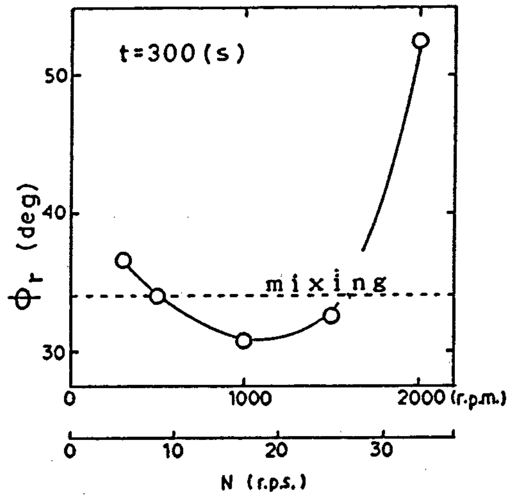


Fig.2 Relationship between angle of repose ϕ_r and rotaion speed N

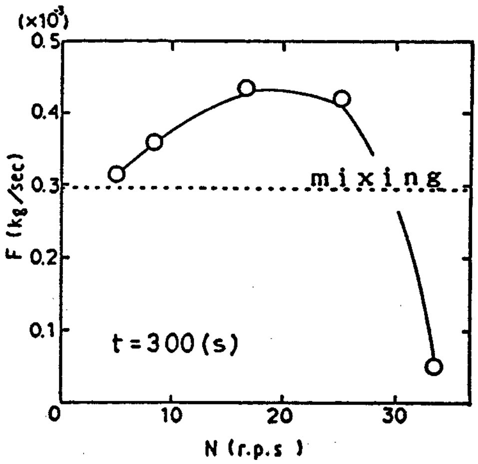


Fig.3 Relationship between outflow speed F and rotation speed N

Uniform flowability with high outflow speed F of composite particles was found at a rotation speed N=16.7 rps and 25.0 rps in Figure 3, which is identical to the results in Figure 2, implying that fluidizer was dispersed on the source particles at optimum conditions at these rotation speeds.

Next, various treatment times were applied at N=16.7 and 25.0 rps, showing the relation between the angle of repose ϕ_r and the treatment time t in Figure 4, the relation between compressibility C and in Figures. 5-1, 5-2, and the relations between outflow speed F and t in Figure 6.

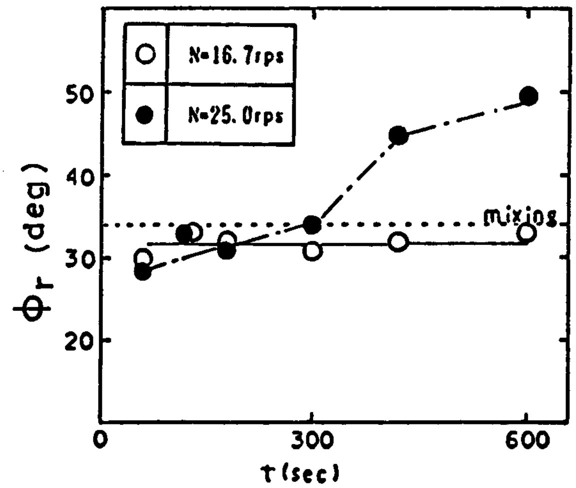


Fig.4 Relationship between angle of repose ϕ_r and treatment time t

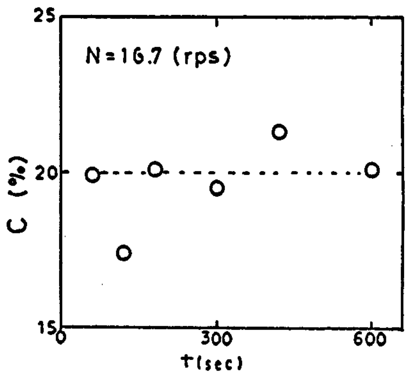


Fig.5-1 Relationship compressibillity C and treatment time t

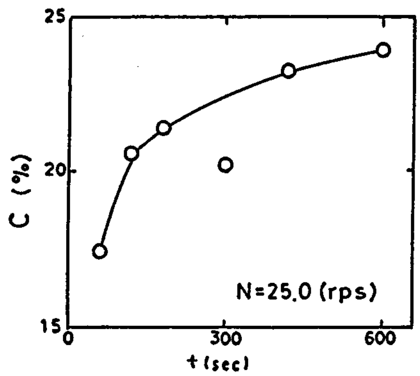


Fig.5-2 Relationship compressibillity C and treatment time t

Figure 4 and Figure 5-2 indicate that flowability of composite particles at N=25.0 rps drops along with the elapse of time t. This can be said also from Figure 6, which indicates a deteriorating flowability of composite particles shown by the decline of F following the increase in t. On the other hand, the physical properties of composite particles at N=16.7 rps remain constant regardless of t size, implying N=16.7 rps possesses the mechanical energy (sharing, compression) to cause the optimum dispersion of primary particles.

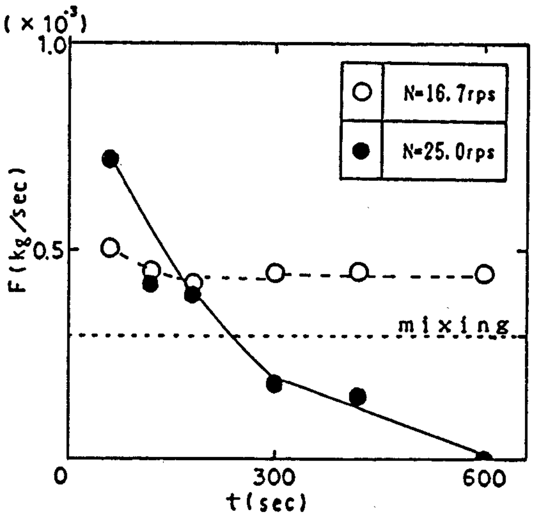


Fig.6 Relationship between outflow speed F and treatment time t

The electrification was examined by an E-spart analyzer to determine the flowability. Figures. 7 to 10 show the E-spart for corn starch, silica, mixed particles, and composite particles. The intersecting point with 0 electrification of corn starch, silca and mixed particles is located at a relatively high position while that of composite particles is low with decresed numbers of inverse polarity (positive electrification). Such a difference in E-spart affects the flowability.

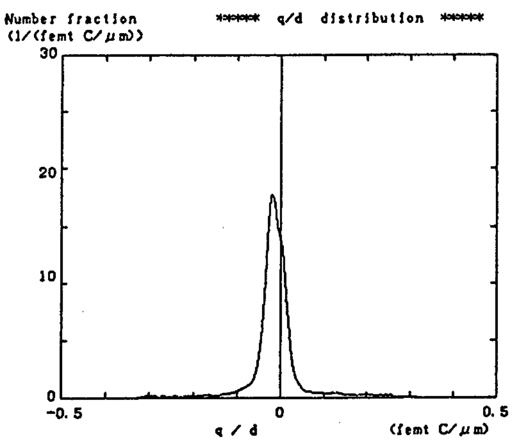


Fig.7 Electrification of corn strch investigated by an E-spart analyzer

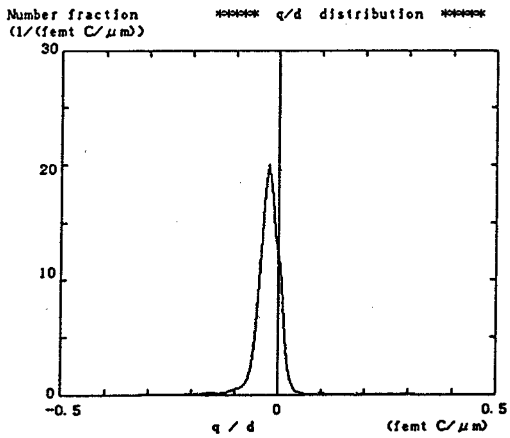


Fig 8 Electrification of silica investigated by an E-spart analyzer

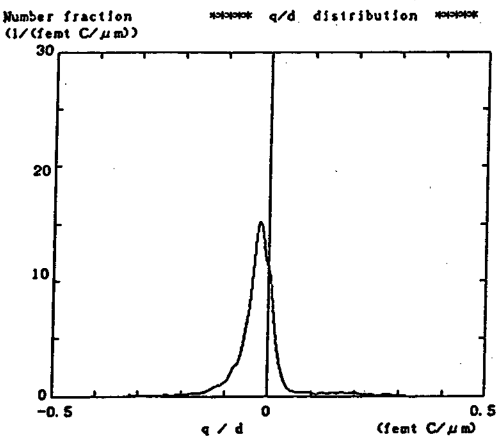


Fig.9 Electrification of mixed particles investigated by an E-spart analyzer

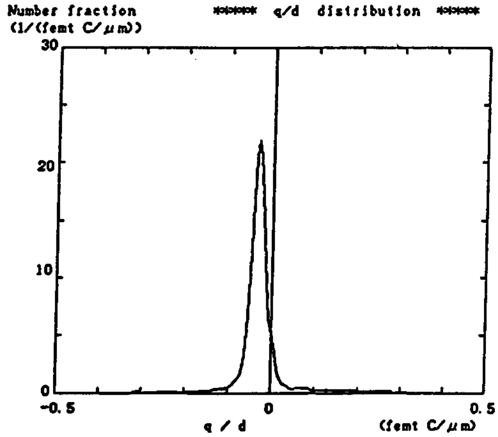


Fig.10 Electrification of composite particles investigated by an E-spart analyzer

Figure 11 shows the relation among electrification Q, the ratio of minus electrification R, and treating time t. Accordingly Q and R drop in proportion to the increase in t, implying fluidizer silica on the suface of the corn starch is buried and reduced in the source particles as t increases, further implying that larger electrification retains more fluidizer on the surface of the source particles. Mixed particles carry many silica particles on the surface but they are secondary particles which are not strong enough to compoud sufficiently. Next, the reason for good flowability of composite particles at t=600 seconds despite decreasing silica on the corn starch surface at N=16.7 rps was desired. Then the quantity of silica on the surface of the source particles at t=600 seconds was calculated by presuming that particles are 1. 0 wt% at t=600 seconds and by a rule of three. The sum is as follows.

$$-1.4:1.0=-0.65:x, \ x=0.45\text{wt\%}$$

It is empirically known that silica fluidizer of 0.45wt% gives sufficient flowability, implying superior flowability is available at N=16.7 rps and t=600 seconds as the fluidizer still binds on the surface of the source particles. No deformation of and compounding between source particles took during the course of the experiment.

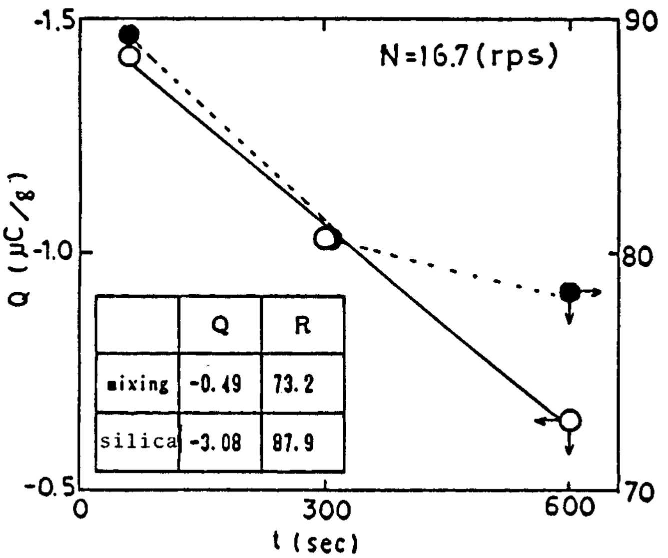


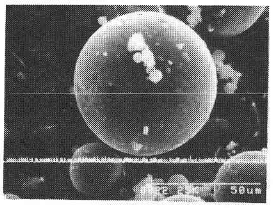
Fig.11 Relationship between electrification Q and ratio of minus electrification R

4. Development of conductive particles by dry coating process

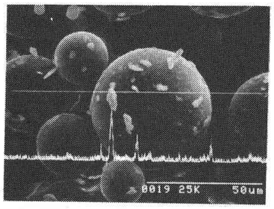
Along with the progressive development of informed society, electromagnetic interference (EMI) has become a social issue in Japan. EMI is caused by the adoption of plastic housings for electronic equipment and measurement instruments. Plastics are easy to form, lightweight, and mass produced. There are two approaches to endow conductivity and electromagnetic wave shielding to plastics. One is to coat conductive film (so-called “surface treatment”) and the other is to weave conductive filler into the resin (so-called “composite particles”). We have been engaged in R&D for the latter, and have published reports on our results. There are various ways to coat conductive film such as coat conductive paint, electrolytic plating, vacuum evaporation, of which conductive paint is the most popular. In this paper, reference is made to dry coating of conductive powder on resin particles, which saved processing steps and reduces the coat compared with conductive painting while conductive composite particles (compound of dispersed particles) are obtained.

PMMA particles of an average 50 μm gauge were adopted as the core resin while fine powder coated with tin oxide or silver was adopted as conductive.

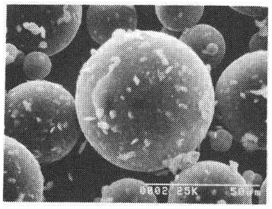
The conductive particle gauge/core particle gauge ratio was in the 8 to 10 range. Specific quantities (0.1kg) of core resin (not premixed) and conductive powder were placed in the casing of Ang Mill which was kept at 33.3 rps and treated for about an hour. The subject material was sampled every 600 seconds to find the coating condition of the conductive powder to the resin particle varying in accordance with the elapse of treatment time. Various additive ratios of conductive powder were applied in the experiment. Figure 12 shows the resin particles coated with silver. The coating (dispersion) condition of the conductive fine powder on the particles improves as treatment time t increases, thus producing particles with superior conductivity. Composite conductive particles in the semiconductor and condutor domains were obtained by applying different specific cubical resistances, powder additive ratios, and treatment times. The particles in the semiconductor domain are used as inhibitors of static electricity while those in the conductor domain are used to control the electromagnetic shield. With such particles, parts more functional that injection molded products can be made.



t=600 seconds



t=1800 seconds



t=3600 seconds

Fig.12 Investigation of particles coated with silver

5. Conclusions

(1) Composite particles with superior flowability are obtained by dispersing fluidizer in optimum uniformity on the source particles.

(2) Particles of superior flowability are not obtained from either weak mechanical energy (shearing, compression) causing bad dispersion of the fluidizer or excessive energy causing fuidizer to bury in the source particles.

(3) Composite particles with superior flowability are obtained by mechano-fusion (Ang Mill) which disperses fine powder better than a high-speed mixer.

(4) Flowability is assessed by checking the bonding condition of the fluidizer on the source particles.

(5) Conductive particles from the semiconductor domain to the conductor domain were obtained by dry coating conductive fine powder on resin particles.

Nomenclature

C : compressibility, %
N : rotation speed, rps
t : treatment time, sec
F : out flow speed, kg/sec
Q : electrification, μc/g
R : ratio of minuselectrification, %
ϕ_r : angle of repose, deg

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