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Powder Mixing and Kneading

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Relationship between powder properties and mixing state, which is important in a field of powder mixing, was investigated.

It was clear that the mixing state (the degree of mixing) in a fixed-type mixer was hardly affected by particle size ratio, density ratio and internal friction coefficient. In case of tumbling mixer, mixing conditions obtaining a satisfactory mixing state of mixture composed of different powder properties were suggested, and it was indicated that the mixing of this type powder system is promoted by convective mixing and shearing mixing.

As an example of kneading, kneadings of magnetic recording materials were employed. The relationship between the kneading and the dispersion state as well as their evaluation methods were discussed.

The state of kneading could be appreciated by observation of coating state of binder on the component particles and the state of dispersion could be evaluated by square ratio and orientation ratio. A satisfactory kneading state contributed to dispersion of magnetic powder materials and ensured high-grade videotape.

In conclusion, it can be said that a performance of videotape depend on its kneading state.

1. Introduction

Powder mixing, kneading and dispersion processes are widely used in the chemical, pharmaceutical, food, ceramics and other industries to blend the raw materials, to make uniform quality products and other purposes.

Powder mixing is a process to uniformly disperse two or more types of dry or slightly wet (pendular state) powder.

In dispersion, the material is moistured with a specific liquid so as to be dispersed uniformly in the liquid (in capillary or slurry state).

Kneading is defined as a process where a small quantity of liquid (binder) is added to the powder to obtain materials of equal quality by coating each component particle with the liquid, or a process where a small quantity of solid or liquid additive is kneaded forcibly into a highly viscous material in sol state. The main objective of mixing and kneading processes is to evenly distribute the component particles. In addition to the above objective, these processes are widely applied for granulation, acceleration of chemical reaction and drying, or their combination. Hence, various mixing and kneading conditions are required according to the respective purposes. The materials to be processed and their properties differ quite largely from each other. The quality of mixing and kneading affects the product quality.

Various types of mixers and kneaders have been developed to satisfy each specific

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purpose and material.

As far as powder mixing is concerned, mixture ratio measuring and its indicating techniques have been developed.

Mixing mechanism, as well as the relation between each mixes and its optimum operating condition, have been also made clear. However, the relation between the properties of powder and the degree of mixing has not yet analyzed completely.

In case of kneading, on the other hand, since it is difficult to understand the rheological property of the kneaded material and to evaluate the degree of kneading, less reports have been presented than that on powder mixing.

This article describes and discusses the relation between the properties of powder and the performance and operation of mixer at first. Then the kneading of magnetic recording materials is taken as an example. A method for evaluating the degree of kneading and dispersion, as well as the correlation between kneading and dispersion states, will be also discussed.

2. Relation between Powder Properties and the Degree of Mixing¹⁻⁷⁾

The largest objective of powder mixing is to prevent the component materials of different particle sizes, densities and flowabilities from segregating, and thus obtaining a desired quality of mixture. Therefore it is decisively effective, in mixer type selection, proper mixer design and optimum processing condition determination, to investigate the relation between powder properties and the degree of mixing in advance.

There are two types of powder properties that affect the degree of mixing; powder properties as a single particle and a bulk powder. Particle sizes and their distribution, density and shape belong to the former. Apparent density, angle of repose, flowability and aggregation characteristic belong to the latter.

Introduced hereafter are the results of a few studies made on the performance of mixers in relation to some of the above properties.

When powder of different particle sizes or densities are mixed by a horizontal (revolution) cylinder type mixer at a low revolutionary speed, a segregation zone is produced inside the mixer (see Fig. 1). The reason is that a group of small but dense particles is segregated into the central zone of the particle layer when they pass through the mixing zone, because of their penetration force and other effects. The character-



Fig. 1 Illustration of segregation in mixer.

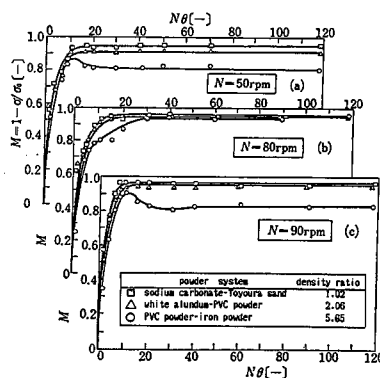


Fig. 2 Characteristics of mixing curves.

istics of mixing during the above segregation process are shown by a curve with o-marks in Fig.2 (a). As can be seen from the figure, the final degree of mixing cannot be obtained as desired.

It has been already known that this type of segregation also appears in V-type, cone type and double cone mixers. The degree of segregation depends on the ratio of particle sizes, the ratio of densities and the mixing speed, and affects the degree of mixing seriously.

In the case where the powders are mixed while they move down by gravity, such as in tumbling mixers (including horizontal cylinder type, V-type, cone type, double cone type, etc.), they tend to be segregated and hence decrease the mixing state, if they have a low coagulation force and both of their particle size ratio and density ratio are nearly 1.5 or less. Therefore, the final degree of mixing cannot be obtained as originally desired.

The segregation zone inside the mixer has to be broken to minimize segregation of the particles. One of the measures is to feed a mixing aid (such as rubber balls) into the mixer so as to disturb the particle flow pattern and to accelerate radial mixing.

Another example is to increase the mixing speed so as to lessen the movement of dense or smaller particles toward the central part of the mixing zone (See Fig. 2 (b)). Thus, it is important to release the particles from gravitational influences as much as possible by disturbing a stable powder circulating pattern and a long mixing zone being formed.

A tumbling mixer with a rotary mixing blade or baffle plates is effective for mixing particles of remarkably different properties.

Figure 3 shows the result of an investigation in which the effects of particle size on the standard deviation of the degree of mixing were measured for various types of mixers. Smaller standard deviation means better degree of mixing. As can be seen from Fig. 3, the degree of mixing in the planetary screw mixer and ribbon mixer (Fig. 4) is little influenced by the particle size.

This feature was also found in mixing particles of different densities.

The reason for the least influences of particle size and density on the degree of mix-

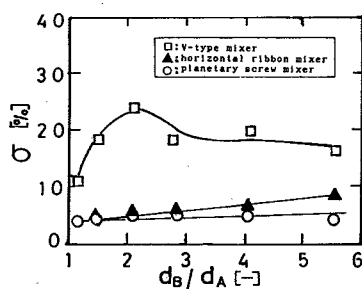


Fig. 3 Relationship between standard deviation and particle size ratio.

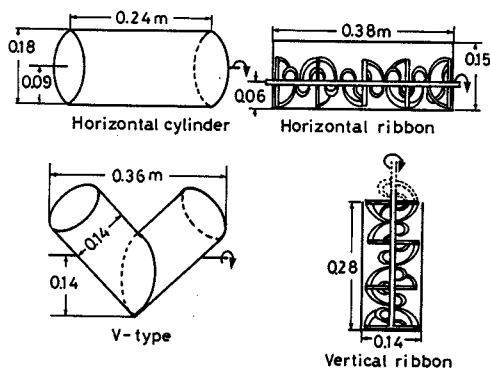


Fig. 4 Experimental mixers.

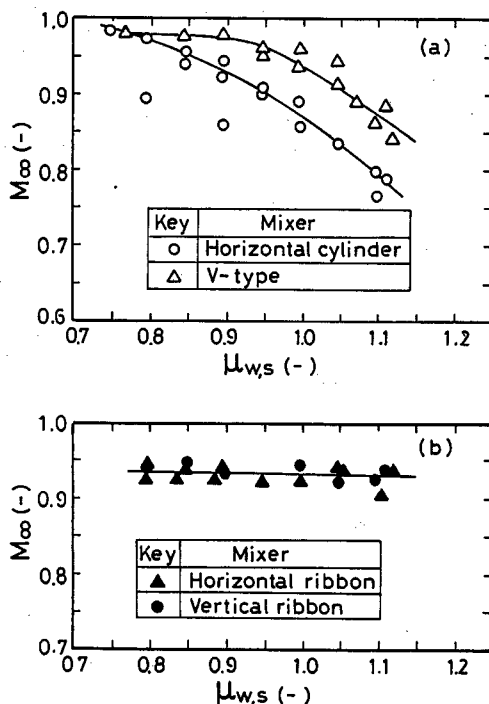


Fig. 5 Effect of internal friction coefficient on final degree of mixing.

ing in tumbling mixers, such as ribbon screw mixer and planetary screw mixer, is considered to be due to the fact that this type of mixer mainly utilizes shearing and circulation of the particles as the mixing mechanism which shorten the mixing zone.

Behavior of the particles in a mixer is considered to be influenced by not only the primary properties, such as particle size and density, but also the secondary properties concerned with flowability. One of the factors which affect the flowability is the angle of repose.

The smaller the angle of repose, the better the flowability. Using a V-type mixer and horizontal cylinder type mixer, it was clarified that a highly flowable powder increases the mixing rate.

In addition, a report describes that powders having high Carr's flowability indexes can be mixed at higher rates.

In addition to the angle of repose, internal friction coefficient is used as an index for evaluating the flowability of powder. The internal friction coefficient is obtained by a surface shear test^{8,9)}. It has been found that the smaller the internal friction coefficient, the higher and better the flowability. Figure 5 shows the effect of the internal friction coefficient $\mu_{w,s}$ of two component type mixture on the final degree of mixing M_{∞} in various types of mixers. As can be seen from the figure, the final degree of mixing in the horizontal cylinder type mixer and V-type mixer decreases when the internal friction coefficient increases. This fact indicates that better final degree of mixing cannot be expected from larger internal friction coefficient.

To the contrary, the final degree of mixing in ribbon screw type mixers is hardly affected by the internal friction coefficient. It has been also made clear that the mixing

rate in a ribbon screw type mixer is hardly affected by the internal friction coefficient.

Hence, it is important to select a mixer combined with forced convection mixing and shearing effect for mixing two component type powders having a high internal friction coefficient.

3. Kneading of Magnetic Recording Materials^{9,10)}

Much higher density and performance floppy disks, video tapes and other magnetic recording media are required to meet the demands of the current information-oriented society. To meet the above requirement, magnetic particles should be powders finely. In addition, each particle of the fine magnetic powder should be dispersed uniformly for high density packing of coated layer and for even magnetic zone. However it is very difficult to make magnetic tapes of superior characteristics by uniformly dispersing agglomerative fine particles with large surface areas. As a measure to overcome the above difficulty, a kneading process is arranged before the dispersion process in a magnetic tape production line. The main objective of the kneading process is to coat each of the fine particles with a binder, while the dispersion process is to loosen the kneaded powder, thus obtaining a high dispersion magnetic coating.

A method for evaluating the kneaded and dispersed conditions of magnetic powder will be discussed in this section. A test was conducted to find the optimum conditions for making superior quality video tapes. The results will be also described.

Figure 6 shows the flow of a continuous kneading process. The kneader is of self-cleaning type in which two shafts rotate in the same direction at the same speed. The kneading chamber is 0.661 m in axial length and $1.16 \times 10^{-3} \text{ m}^3$ in volume. The kneader used for the test is provided with an automatic measuring system. This system

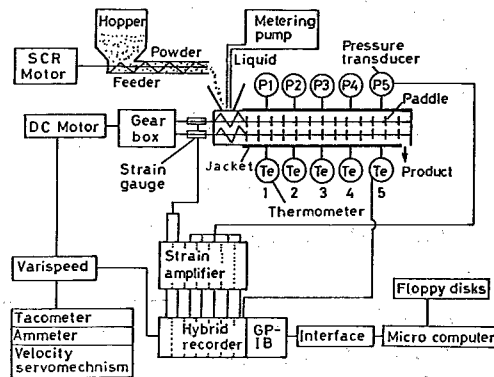


Fig. 6 Flow chart of continuous kneading.

Table 1 Powder samples for magnetic tapes

Sample	Symbol	d_p [μm]	ρ [kg/m^3]
Magnetic powder	M	0.1 ~ 0.5	5260
Carbon black	C	0.01 ~ 0.1	2100
Alumina	A	0.1 ~ 0.5	4220

measures automatically the temperature and pressure distributions on the kneading chamber wall, as well as the mixing torque and speed.

Three types of paddles were used to accelerate the kneading rate by shearing, pressurizing and expanding the powder. Table 1 lists the powders used for the test. As the kneading liquid (binder solution), an organic solvent (three component solvent consisted of MEK, toluene and cyclohexane) containing a binder resin (two component type consisted of cellulose nitrate and polyurethane) was used.

For the test, magnetic powder(M), magnetic powder/alumina powder compound (MA), magnetic powder/carbon black powder compound (MC) and magnetic powder/alumina powder/carbon black powder compound (MAC) were kneaded with an equal content of kneading liquid (volume of liquid $V = 0.56 \times 10^{-3} \text{ m}^3/\text{kg}$, quantity of binder resin $B = 20 \text{ wt}\%$).

Figure 7 and 8 show the results of mixing torque (T) measurement. As can be understood from Fig. 7, the mixing torque for M and MA fluctuates sharply. The powder compound kneaded with such fluctuating mixing torque was partly soft and partly hard, and its glossiness was not even. Thus it was hard to obtain continuously a kneaded powder of uniform quality.

In contrast to M and MA, Fig. 8 shows that the mixing torque for MC and MAC reached a steady value (T_s), after having passed through a transient condition (max. T_{max}).

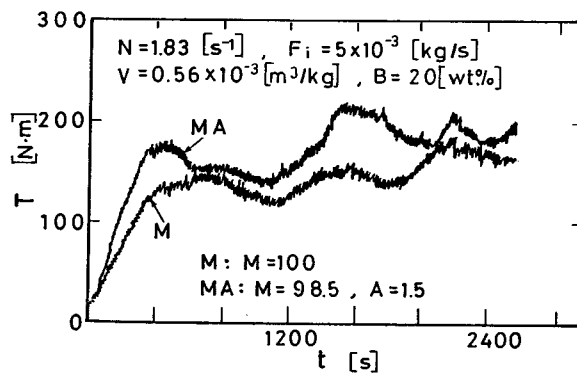


Fig. 7 Mixing torque on M and MA.

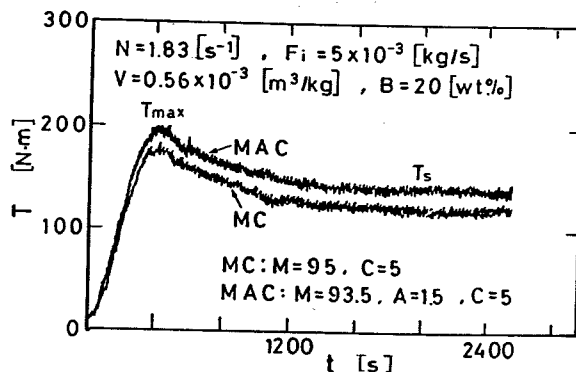


Fig. 8 Mixing torque on MC and MAC.

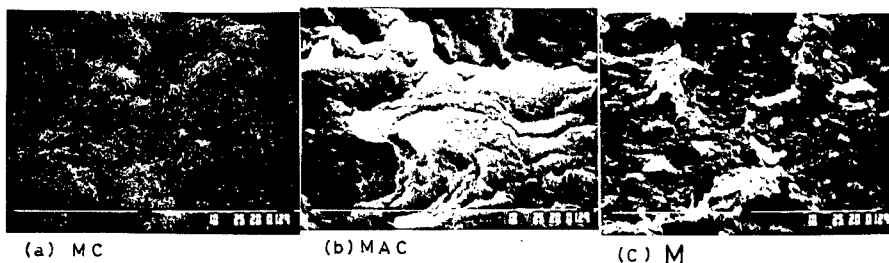


Fig. 9 SEM view of kneaded material.

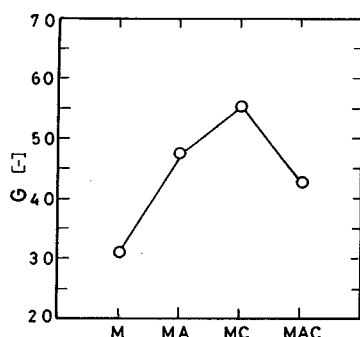


Fig. 10 Measurement results of glossiness.

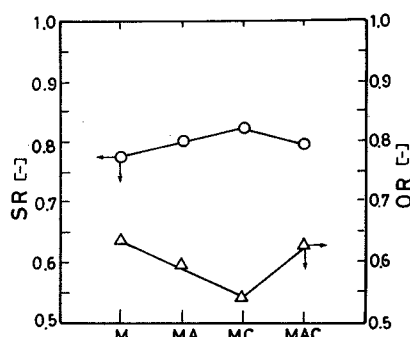


Fig. 11 Square ratio and orientation ratio.

The mixing torque itself does not fluctuate sharply.

Moreover, the steady mixing torque T_s is smaller than that of other compounds. The reason for little fluctuated mixing torque and smaller T_s value for MC is considered to be due to the lower interparticle adhesion and better flowability of this type compound.

When the MC-compound is kneaded, the surface of the resulting cake was smooth and glossy, indicating a favorable degree of kneading.

Figure 9 shows an example of observations of kneaded powder through a scanning electron microscope. In the MC-compound, each particle is completely coated with the binder resin as shown in Fig. 9 (a), resulting in a satisfactory-degree of kneading. In case of MAC-compound, to the contrary, large aggregated bulks (white color) of the binder resin are observed. Hence, no desirable degree of kneading can be expected [see Fig. 9 (b)].

Aggregated bulks of binder were also observed in the M-compound, as shown in Fig. 9 (c).

As has been described above, the magnetic powder/carbon black power compound offers a preferable degree of kneading as a magnetic tape compound.

Subsequent to the above test, the degree of dispersion of magnetic powder was evaluated. For the evaluation, kneaded powder was diluted and dispersed to form a coated layer. The glossiness and magnetic properties of the coated layer were investigated.

The result of coated layer glossiness (G) measurement is shown in Fig. 10. As can be seen from this figure, the MC-compound shows the largest glossiness and smooth coating.

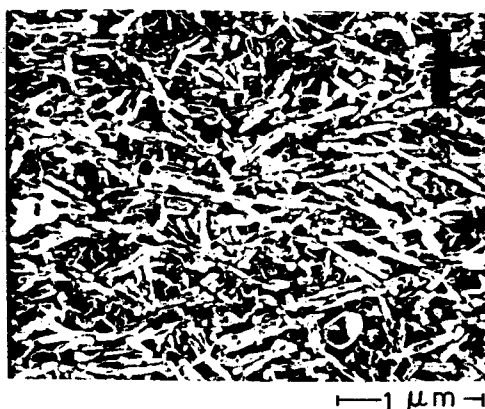


Fig. 12 SEM view of videotape surface.

That is, the MC-compound disperses the magnetic powder desirably in the coated layer. Hence, this type of powder compound provides a preferable degree of kneading. In other words, when a kneaded powder with a desirable degree of kneading is diluted and dispersed, the resulting coated layer contains least aggregate but uniformly dispersed magnetic powder.

Figure 11 shows the magnetic properties; square ratio (S.R) and orientation ratio (O.R) of each compound.

As can be understood from the figure, the MC-compound has superior magnetic properties; smaller O.R and larger S.R. Hence, superior quality magnetic tapes can be made by kneading the MC-compound, because the magnetic powder is dispersed uniformly and filled densely in the coated layer.

Figure 12 shows an example of the surface of a video tape having a superior magnetic quality. As is seen from the figure, each magnetic particle is coated desirably with the binder (white color) and is distributed uniformly in the coated layer in a form of the primary particle unit.

As described up to know, the degree of kneading can be evaluated by observing the powder coated with a binder or measuring the mixing torque. The degree of dispersion can be evaluated by measuring the glossiness and magnetic hysteresis curve (S.R. and O.R).

4. Conclusion

A method for quantitatively evaluating the degree of kneading based on various data should be established to bring the kneading process from experience-based technique into science-oriented technique¹¹⁻¹⁵. It is essential for effective mixing and kneading to study the mixing and kneading mechanisms of various types of mixers and kneaders. With the progress of studies in this field, more effective design and selection of mixers and kneaders as well as determination of optimum processing condition can be made. The objective of mixing and kneading should be made clear. Properties of particles should be determined. And the flowability of solid-liquid state materials and highly viscous materials has to be investigated. In addition, particle size distribution, surface tension and wetting of the kneading liquid should be measured accurately.

Kneading process should be analyzed in the future, on the basis of these factors.

Mixing and kneading processes should not be understood as a unit process of powder treatment process, but should be grasped as series of powder treatment process, in order to introduce a system engineering technology into this field in the future.

Nomenclature

A :	alumina
B :	binder content, wt. %
C :	carbon black
dB/dA :	particle size ratio, —
d_p :	particle size, μm
F_i :	feed rate, kg/s
G :	glossiness, —
M :	degree of mixing or magnetic powder, —
MA :	magnetic powder-alumina
MAC :	magnetic powder-alumina-carbon black
MC :	magnetic powder-carbon black
M_∞ :	final degree of mixing, —
N :	rotation speed, rpm or s^{-1}
$N\theta$:	total number of rotations, —
T :	mixing torque, $N\cdot m$
T_{max} :	Maximum mixing torque, $N\cdot m$
T_s :	steady mixing torque, $N\cdot m$
t :	time, sec

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