Continuous Kneading of Electric Conductive Resin for EMI Shield Material

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Yoshihisa MIZUNO*, Keijiro TERASHITA** and Kei MIYANAMI**

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In continuous kneading of composite electric conductive resin, its conductivity is influenced by dispersion state of fillers in the matrix resin. We performed these experiment, changing feed rate of raw material mainly, and specific resistance were measured to evaluate the electric conductivity. After kneading, share force among kneading and the length distribution of fillers in the matrix were measured, and employed for evaluating the dispersion state of fillers. RTD function of objective material in the kneading vessel were also employed to analyze these phenomena. From these reseults, we can clarify that the relation between conductivity and dispersion state of fillers, and why these phenomena yield.

1. Introduction

Recent years, electromagnetic interference (EMI), which results in misoperation or accident by noise that generated from electronic instruments, has been serious problem as an industrial pollution.

EMI is caused, so that advancing of electronic industries, and housing of electronic instrument changes metals to plastics which characteristics are that lighter than metals, that has mass productivity, and the electromagnetic waves passes through. Therefore, we must give the plastics electrical conductivity using any methods.

Till now, the method of forming conductive layer on the surface of plastics is employed mainly. But, we take notice of the method of filling the plastic with electric conductive filler, this material is called composite conductive plastics, from productivity and economical point as EMI shield material.

We have performed kneading of composite conductive plastics using ABS resin as matrix and metalized grass fiber as conductive filler. And, kneading condition, such as, kneading temperature, feeding rate of raw material and revolution speed of kneading paddles, was changed. As a result,we could clear the optimum condution that we got excellent shielding effect. At the same time, we suggested that shielding

* Graduate student, Department of Chemical Engineering, College of Engineering
** Department of Chemical Engineering, College of Engineering

effect was concerned with dispersion state of filler in the matrix resin[1,2]. But, relation between dispersion state of filler and shielding effect or kneading condition was not made clear enough.

In this study, we perform the kneading of electric conductive resin for EMI shielding material to get the relation described above, and feed rate were changed mainly. We estimated the dispersion state of fillers from distribution of them, breakage state of fillers in matrix resin, and flowability of objective materials in the kneading vessel.

2. Experimental

The continuous kneader and measurement system used in this work is illustrated in Fig. 1.

The kneader has two rotation rods that rotates the same direction. Feeding screws or kneading paddles are able to be fixed on each rod.

The vessel of kneader is heated with band heater. Raw materials are fed with vibrating feeder. The temperature of objective material and heater are measured with thermocouple, and measuring data during kneading is monitored and recorded using personal computer. The kneading torque is also monitored and recorded.

The raw materials wear shown in Table 1. ABS resin is employed as matrix resin, and glass fiber coated with three metalic layer, nickel-copper-nickel, as electrical conductive filler.

Kneading experiment was performed as follows: after confirmed that temperature of the kneading vessel reaches stated amount and steady state, only ABS resin, as matrix resin, was fed to kneader. For a while, about 1200 seconds, kneading state reacehes stable state. At that time, feeding was switched to mixture of ABS resin and filler which had been premisxed. And then, the kneading materials were sampled

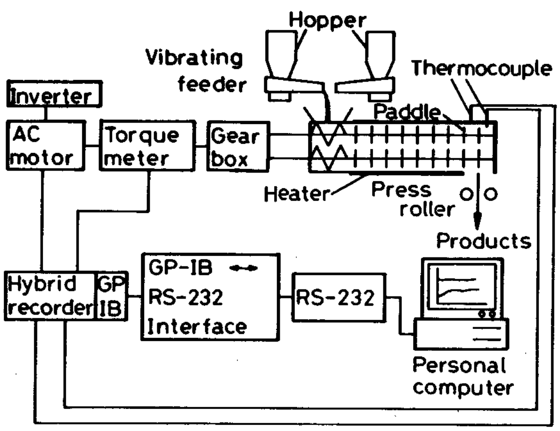
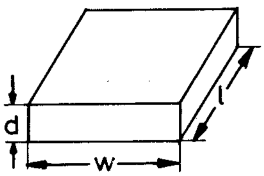


Fig. 1 Schematic diagram of continuous kneading.

Table 1 Properties and shapes of raw materials

Properties and shapes \ Materials		ABS resin	Metarized glass fiber
Glass transition point	(k)	390	—
Density	(kg/m^3)	1040	3600
Shapes		Pellt	banding 2 300 of fibrs
Diameter	(m)	2.5×10^{-3}	13×10^{-6}
Length	(m)	3×10^{-3}	3×10^{-3}



$$\rho_{v_i} = R\cdot(d\cdot w)/l$$

$$C\rho_v = S/\bar{\rho}_v,\ S=\left[(1/n)\sum_{i=1}^{n}(\rho_{v_i}-\bar{\rho}_v)^2\right]^{1/2}$$

$$\bar{\rho}_v = \left[\sum_{i=1}^{n}\rho_{v_i}\right]/n$$

Fig. 2 Illustrationof moulded electric conductive resin.

at fixed time intervals, 30 or 60 seconds, and moulded 5 mm thickness by press roller. The moulded kneading materials were cut in square, 18×18 mm, and calculate the specific resistance measuring the resistances and the size of kneading materials closely (Refer to Fig. 2)[1,2].

The shear force acting on paddles, mean length of glass fibers in the matrix resin, and residence time distribution of objective material were employed to clarify the phenomenon. The shear force acting on kneading paddles was calculated in Eq. 1. Mean length of glass fibers was calculated to

$$\tau = Tq/Vh \tag{1}$$

Tq: Kneading torque, *Vh*: Hold up volume of objective materials. Average the length of 400 fibers that was separated from matrix resin to solve the resin. Residence time distribution was calculated to perform tracer test described later.

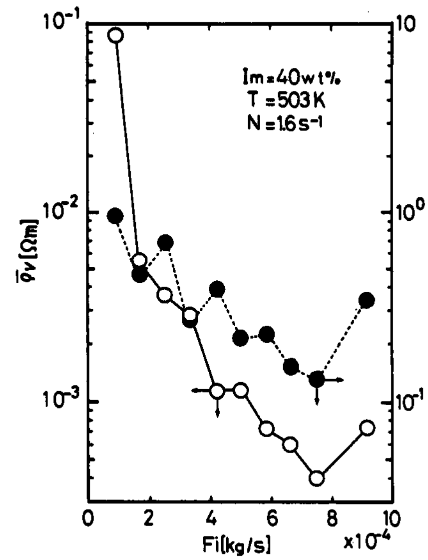


Fig. 3 Mean specific resistance $\bar{\rho}_v$, variation coefficient of specific resistance C_{ρ_v} versus feed rate *Fi*.

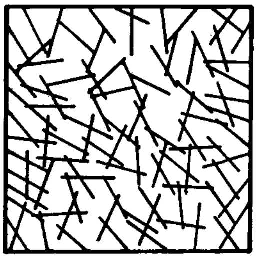


Fig. 4 Illustration of electric circuit by conductive fillers in the matrix resin.

3. Results and Discussion

Figure 3 presents mean specific resistance of kneading material, $\overline{\rho_v}$, and its variation coefficient, C_{ρ_v}, under several feed rate, *Fi*, conditions.

It shows that the mean specific resistance increases as the feed rate decreases, in other words, the electric conductivity decreases as the feed rate decreases, but at $Fi = 9.33 \times 10^{-4}$kg/s, the mean specific resistance decreases. The variation coefficient increases as the feed rate decreases. If value of variation coefficient is small, kneading of electric conductive resin is performed keeping stability.

Figure 4 is illustrated the electrical circuit formed by conductive filler in the matrix resin. From this illustration, it is said that the electric conductivity of kneading material is decided by dispersion state of filler. So, we evaluate the dispersion state of filler with shear force acting on kneading paddles and breakage state of filler, in order to explain this phenomenon.

Figure 5 presents shear force acting on paddles, τ, and mean length of fillers which shows breakage state of filler, L_F, as a function of feed rate. Shear force has the peek at $Fi = 5.00 \times 10^{-4}$kg/s, and has large values at the region of high feed rate, in other words, the filler is dispersed satisfactory at this region. The length of electric conductive filler in the kneading material decreases as feed rate decreases.

From these results, at the region of high feed rate, the electric conductive fillers are dispersed satisfactory in the matrix resin, and broken by kneading is small, so that we can get the electric conductive resin which has excellent electric conductivity. But, the region that the feed rate is too high, the fillers are not dispersed satisfactory, electric conductivity of kneading material is less increased.

To consider the phenomena that appeared Fig. 5, we evaluate the flowability in kneading vessel with residence time distribution (RTD) function of objective material. RTD function was obtained from impulse answer test with glass fiber as tracer. Mean residence time and hold up volume of objective material are also obtained to apply the dead space model[3].

Figure 6 presents the relation between the mean length of fillers, L_F, in the matrix resin and mean residence time of objective material, . Mean length of fillers are kept long when mean residence time is short. Considering the result of Fig. 5, when the feed rate is high, mean residence time becomes short, and the opportunity that kneading paddles give shear force the fillers directory is reduced. As the reseult the

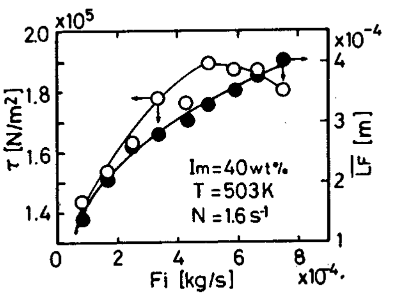


Fig. 5 Shear force τ, meanlength of filler L_F versus feed rate Fi.

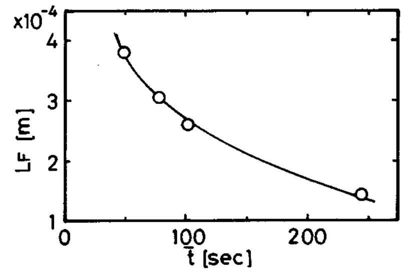


Fig. 6 Relation between meanlength of filer after kneading L_F and mean residence time $\bar{t}$.

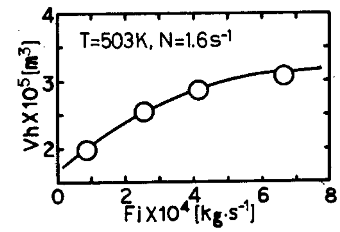


Fig. 7 Hold up volume Vh versus feed rate Fi.

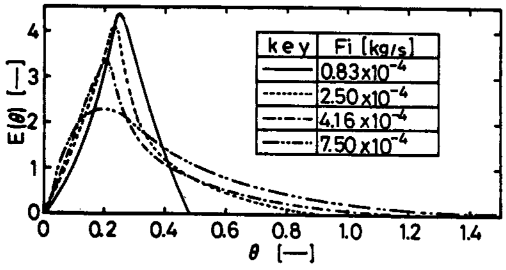


Fig. 8 RTD function $E(\theta)$ under several feed rate conditions.

fillers are kept long.

Figure 7 presents the relation the hold up volume of objective materials in the kneading vessel, Vh, between feed rate of raw material, Fi. The hold up volume decreases as feed rate decreases. When hold up volume is large, kneading force is conducted to the objective material efficiently, as the results share force has large value.

Figure 8 presents the RTD function $E(\theta)$, under several feed rate conditions. When the feed rate decreases, flowability of kneading material approaches perfect mixing, and when the feed rate increases, flowability aproaches plug flow. This kind of flowability is important because that decides stability of kneading. Generally, the closer flowability approaches perfect mixing, the more property of kneading material (electric conductivity) stabilizes.

From this point of view, we can explain the relation between variation coefficient and feed rate shown in Fig. 3. That is, the region which feed rate is high, flowability of objective material approaches perfect mixing, so that influence of variation at the entrance, such as, small variation of feed rate or kneading temperature, is reduced among kneading in the vessel. Besides, in the field of powder mixing, plug flow is required[4], but in this study, it is not followed that judgement, because we must consider breakage state of the electric conductive filler, and stability of kneading.

4. Conclusion

(1) Electric conductivity of composite conductive resin is controlled by distribution of electric conductive filler in the matrix resin and breakage state of the filler.

(2) Distribution state of the filler, length of filler, and stability of property which is electric conductivity are concerned with flowability of objective material that flows in the kneading vessel.

Nomenclature

C_{ρ_v}	= variation coefficient of specific resistance	[−]
d	= thickness of electric conductive resin	[m]
$E(\theta)$	= RTD function	[−]
Fi	= feeding rate of raw material.	[kg/s]
I_m	= filling rate of electric conductive filler	[wt%]
L_F	= mean length of filler after kneading	[m]
l	= length of electric conductive resin	[m]
N	= revolution speed of the rotation rods	[s^{-1}]
n	= number of samples	[−]
R	= resistance of electric conductive resin	[Ω]
S	= standard deviation of specific resistance	[Ωm]
T	= kneading temperature	[K]
Tq	= kneading torque	[J]
t	= time	[sec]
$\bar{t}$	= mean residence time	[sec]
$\tilde{t}$	= space time	[sec]
Vh	= Hold up volume of kneading material	[m^3]
W	= width of electric conductive resin	[m]

Greek symbols

θ	= dimensionless time $(=t/\tilde{t})$	[−]
$\overline{\rho_v}$	= mean specific resistance	[Ωm]
ρ_{vi}	= specific resistance of its sample taken with time in continuous kneading	[Ωm]
τ	= shear stress acting on paddles during kneading	[N/m^2]

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