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Manufacture of EMI Shield Material by Continuous Kneading with Fragile Fibered Fillers: Influence of Revolution Speed of Paddles on Electric Conductivity

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In continuous kneading with ABS resin and electric conductive fillers (metal coated glass fibers) for manufacturing EMI shield material, the influence of revolution speed of paddles on the electric conductivity and shielding effectiveness of the material was discussed. It was estimated from the dissolution state of bunddled fillers and the breakage state of fillers after kneading.

From the results, it was clarified that the electric conductivity and shielding effectiveness enhanced with decreasing revolution speed and this relates with the dissolution and breakage state of fillers according to revolution speed.

And the homogeneity of productions in continuous kneading was estimated from the variance which calculated from the RTD of fillers. As the results, homogeneous productions could be obtained under lower revolution speed.

1. Introduction

Recent years, with the progress of precision electronical instruments, the electromagnetic interference (EMI), which results in misoperations and accidents by the noise generated from the instruments, has been at issue as an industrial pollution.

The electro magnetic waves intrude through the plasics housing of instrument because it is an electric isolator. Therefore, as a preventive measure, the methods that electric conductive property is given to the plastics have been employed.

The methods are classified into two catagolies.¹⁾ The one is the formation of conductive layer and the other the composition by kneading. The former mainly has been employed till now. The latter is carried out by filling conductive fillers into matrix resin. It demands large amount of fillers to attain high electric conductivity for EMI shield material, so that it is accompanied with the deterioration of intensity, plasticity, economical efficiency and external appearance of production. Therefore, for practical use this problem should be settled.

If the problem be settled, this method will be superior to the former methods on the view points of the simplication of manufacturing process and durability of shield material.

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We have studied on the development of EMI shield materials by this method^{2,3}.

In present work, continuous kneading with ABS resin and nickel coated glass fiber as conductive filler was carried out to manufacture EMI shield material. And the influence of revolution speed of paddles on the electric conductivity and shielding effectiveness of production was investigated. It was estimated from the dispersion state and the breakage state of fillers.

2. Experimental

2.1. Kneading experiments

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

The vessel of the kneader was $0.165m \log and 8.84 \times 10^{-5} m^3$ inner volume except the volume of paddles. The paddles supported by a pair of shaft which rotate to same direction with equispeed by motor, were assembled spirally and each paddle tip of the one side was adjoined to the flank of the other side, so that the paddles promote kneading action with cleaning themselves spontaneously by revolution. It was, so called, a self-cleaning continuous kneader.

A band heater equipped with controller, a thermistor, a torque meter and a transducer were installed at the positions shown in Fig. 1, respectively.

The experimental data measured during kneading were monitored by a displayer and recorded in floppy disk of the microcomputer.

The powder materials used in kneading and their properties are listed in Table 1.

The ABS resin (Acrylonitryl Butadiene Stylene) was employed as a matrix material and the glass fiber coated with nickel as an electric conductive filler.

Kneading experiment was performed as follows: After feeding only ABS resin at the determined feeding rate from the hopper A untill the melting state of ABS resin had

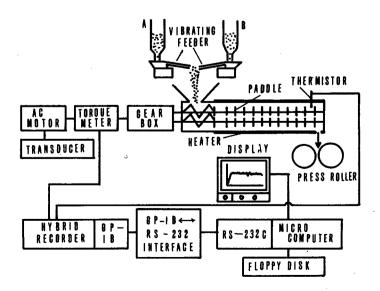


Fig. 1 The continuous kneader and measurement system.

	ABS resin	glass fiber
melting point	422[°K]	_
specific gravity	1.04	3.6
shape	pellet (¢2.5×3mm)	a bundle of 2300 chops (¢l3µm×3mm) coated three layers, Ni-Cu-Ni

Table 1 Properties of materials used for kneading.

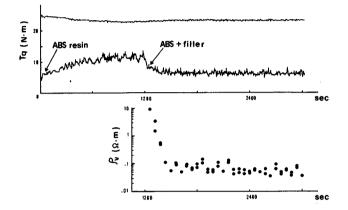


Fig. 2 The variation of torque during kneading and the results of the specific resistance measured.

reached stable state, the feeding was switched to the mixture of ABS resin and filler (hopper B) which had been premixed (see Fig. 2). And then, the kneaded materials were sampled at fixed time intervals, 30 seconds, and moulded in 5 mm thick by press roller.

On the other hand, for measurement of residence time distribution (RTD) of filler, the filler 1 gram was put into inlet as tracer instead of switching the mixture of ABS resin and filler mentioned above and the outputs were sampled at 10 seconds intervals. These were dissolved with a solvent (methylethylketone : toluene = 1 : 1) and fillers were carefully separated and weighed.

The same experiments were carried out under various revolution speeds.

2.2. Measurement of the specific resistance

Materials attained in kneading were cut off in 20×20 mm and the resistance, $R[\Omega]$, of directions A and B respectively were measured as illustrated in Fig. 3. Where, the resistance of aluminium foil was so small that it could be ignored.

From the resistance R, the specific resistance, $\rho_v [\Omega \cdot m]$, was calculated in Eq.(1):

$$\rho_{\nu} = (a \cdot b/d) \cdot R \tag{1}$$

Where, the $(a \cdot b)$ is cross-sectional area, the d distance between the cross sections.

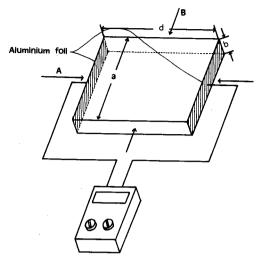


Fig. 3 Measurement of the resistance of the kneaded material.

3. Results and Discussion

3.1. Influence of revolution speed on the specific resistance and the homogeniety of productions in continuous kneading

Figure 4 presents the mean specific resistance, $\bar{\rho}_{\nu}[\Omega \cdot m]$, and the shielding effectiveness, SE[dB], of the materials obtained in continuous kneading as a function of the revolution speed of paddles, $Re\nu$ [rps].

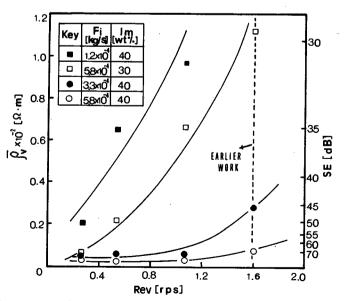


Fig. 4 The mean specific resistance, $\bar{\rho}_{\nu}$, and the shielding effectiveness, SE, as a function of revolution speed of paddles.

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The shielding effectiveness is presented in order to estimate availability of the kneaded material as an EMI shield material and it was calculated from the Schelkunoff's equation⁴⁾:

SE = 50 + 10 log
$$(\rho_v \cdot f)^{-1}$$
 + 1.7 $b (f/\rho_v)^{1/2}$ (2)

Where, the specific resistance, $\rho_{\nu} [\Omega \cdot cm]$, was substituted for the mean specific resistance, $\bar{\rho}_{\nu} (= 1/n\Sigma\rho_{\nu})$, and the frequency of electromagnetic wave, f[MHz], and the thickness of shield material b [cm], were adopted in f = 1000 MHz and b = 0.2 cm, respectively.

It shows that the mean specific resistance decreases as the revolution speed decreases, in other words, the electric conductivity, $1/\rho_{\nu}$, increases with decrease of revolution speed, so that the shielding effectiveness is improved.

Especially, it is worthy of notice that an available shielding effectiveness for EMI shield material, which is demanded about 40-60 dB in general, could be obtained by decreasing revolution speed even in cases of low filling rate of fillers and low feeding rate, in spite that the available shielding effectiveness was obtained under conditions of high filling rate, above 40 wt%, and/or high feeding rate (indicated by broken line in Fig. 4) in our earlier work³⁾.

This fact is very important to improve deterioration of intensity, plasticity, external appearance and economical efficiency of the EMI shield material which results from high filling rate of fillers.

On the other hand, the homogeneity of quality of productions in continuous kneading was appreciated from the variation coefficient of specific resistance, $C_{\nu}[-]$:

$$C_{\nu} = \sigma_{\rho\nu}/\bar{\rho}_{\nu} , \quad \sigma_{\rho\nu} = \sqrt{1/n\Sigma(\rho_{\nu} - \bar{\rho}_{\nu})^2}$$
(3)

Figure 5 presents a relationship between the variation coefficient and the revolution speed of paddles. The fact that the variation coefficient decreases as revolution speed decreases as shown in Fig. 5, implies that the lower revolution speed is, the more homogeneous productions can be obtained in continuous kneading.

It is regarded that the results in Figs. 4 and 5 relate with dispersion state of fillers.

Figure 6 illustrates a conception of transmission paths of electricity through the electric conductive fillers. Comparing A and B in Fig. 6, it can be considered that the electric conductivity depends upon not only dispersion state but also breakage state of fibered fillers.

Therefore, in present work the dispersion state and the breakage state according to revolution speed of paddles are discussed from the residence time distribution (RTD) and the length of fibered fillers, respectively.

3.2. Dispersion of the fibred fillers depending on revolution speed of paddles

At first, let's consider the dispersion process of the bunddled fibred fillers employed in this work. It may be classified into two types: dissolution of bunddled fillers and dispersion of the individual dissolved fillers in the vessel. The former mainly results from shearing of melted matrix resin and the latter from radialmixing and/or lateral mixing.

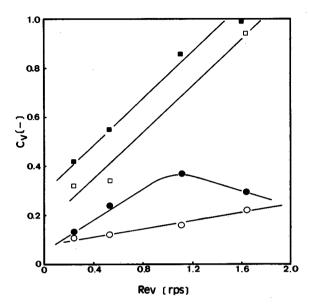


Fig. 5 The fluctuation coefficient, C_{ν} , as a function of revolution speed of paddles. (All keys refer to Fig. 4.)

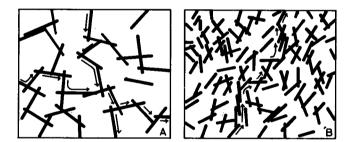


Fig. 6 Illustrations of conceptive transmission paths of electricity through the conductive fillers.

Figure 7 illustrates the residence time distributions in order to appreciate the dispersion states of fillers.

From the RTD curves, the mean residence time, \overline{t} , the variance, σ_{θ}^2 , and the holdup, V_h , were calculated with the Eqs. (4) to (6)⁵⁾:

$$\overline{t} = \int t E(t) dt \tag{4}$$

$$\sigma_{\theta}^{2} = \int \theta^{2} E(\theta) \, d\theta - 1 \tag{5}$$

$$V_h = \bar{t} \cdot Q \tag{6}$$

where, the θ (= t/\bar{t}) is demensionless time and the Q is volumetric feeding rate. Figure 8 plots the mean kneading torque, Tq, measured during kneading as a function of revolution speed. It shows that the kneading torque increases with decrease of revolution speed. From this result, it can be considered that the dissolution of bunddled fillers is

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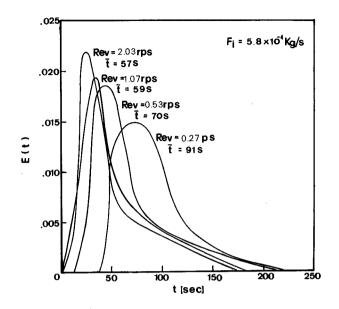


Fig. 7 Residence time distributions according to revolution speed. (The \overline{t} was calculated from Eq. (4).)

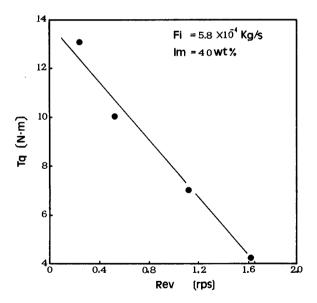


Fig. 8 Relationship between the kneading torque and the revolution speed.

more easily performed under lower revolution speed owing to intensive shear force.

This fact is also confirmed by the Fig. 9 showed photographs of dissolution state of the bunddled fillers after kneading. It shows that the bunddled fillers were discharged before dissolving sufficiently under high revolution speed at which low torque indicates in Fig. 8.

Figure 10 is ploted the holdup, V_h , calculated from the Eq.(6) as a function of revolution speed. The reason why the torque increases with decrease of revolution speed

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can be given from the result of Fig.10. It is due to increasing of the holdup with decrease of revolution speed. Accordingly, it can be said that the increase of holdup is beneficial to dissolve bunddled fillers. On the other hand, the dispersion of dissolved individual fillers by radial and/or lateral mixing can be appreciated from Fig. 11. It presents the

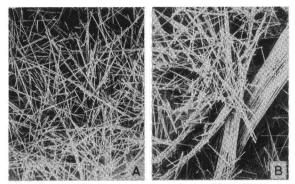
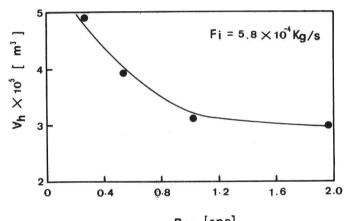


Fig. 9 Photographs of dissolution state of the bundled fillers after kneading. (A: Rev = 0.27 rps, B: Rev = 2.03 rps)



Rev [rps]Fig. 10 The holdup, V_h , as a function of revolution speed.

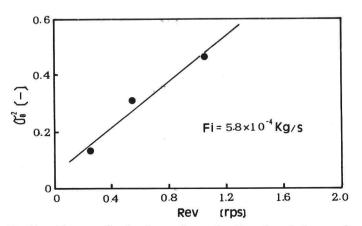


Fig. 11 The normalized variance, σ_{θ}^2 , as a function of revolution speed.

relationship between the variance, σ_{θ}^2 , which calculated from the Eq. (5) and the revolution speed. It shows that the lateral mixing promotes with increase of revolution speed.

This is regarded as an evidence supporting the results in the Fig. 5. Because, in general, it is well known that the variation coefficient of production in continuous kneading is controlled by restraining lateral mixing⁶).

3.3. Breakage of the fibred fillers after kneading depending on revolution speed of paddles

In order to investigate the breakage of fillers, the ratio of the average length of output fillers against that of input fillers, L_{Fo}/L_{Fi} , as a function of revolution speed, *Rev*, is presented in Fig. 12.

It shows that the fibred fillers are severely broken as the revolution speed increases, that is, the length of fibred fillers can be preserved under lower revolution speed.

Here, as the reason of the breakage of the fillers, the number of revolution of paddles and the residence time of fillers can be considered.

Figure 13 presents a relationship between the L_{Fo}/L_{Fi} and the total number of revolutions during kneading, N. Where, the N is calculated from the Eq.(7):

$$N = Rev \cdot \overline{t} \tag{7}$$

From the Fig. 13 it can be realized that the breakage of fillers is able to restrain by means of not merely the revolution speed or the mean residence time but the total number of revolutions during residence time.

To diminish the value of N in Eq.(7), small value of the Rev and the \overline{t} are required. However, these two conditions cannot be satisfied at the same time because the residence time is prolonged by diminishing revolution speed as shown in Fig. 7. Judging from the results discussed in our earlier and present works, both of lower revolution

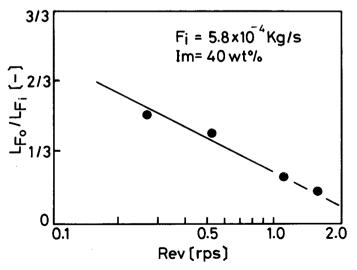


Fig. 12 Relationship between the breakage of fillers, L_{Fo}/L_{Fi} , and revolution speed.

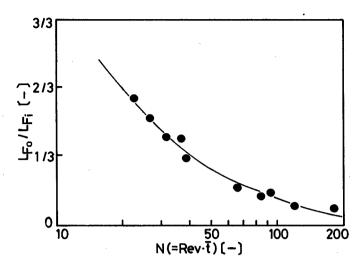


Fig. 13 Relationship between the breakage of fillers, L_{Fo}/L_{Fi} , and the total number of revolutions during residence time, N.

speed and higher feeding rate are recommended as kneading condition for satisfying the conditions. Further more, these conditions have the holdup enhanced.

From this result, together with the result of dissolution of bunddled fillers mentioned above, the phenomena that electric conductivity increases with decrease of revolution speed as given in Fig. 4 can be explained.

4. Conclusion

The electric conductivity of kneaded material in continuous kneading could enhance by decreasing revolution speed of paddles.

Especially, even in cases of low filling rate and/or low feeding rate in which unsatisfactory shielding effectiveness had attained in earlier work, satisfactory shielding effectiveness for EMI shield material was obtained by decreasing the revolution speed. This fact suggests the possibility of high electric conductivity even with low filling rate of filler. It is very important to improve the deterioration of EMI shield material which result from high filling rate of fillers.

The relationship between electric conductivity and revolution speed was estimated from the dissolution state and the breakage state of bunddled fillers according to revolution speed. Under lower revolution speed the bunddles of fillers were well dissolved as well as long fibred fillers were preserved after kneading.

And it was clarified that the breakage of filler relates with the total number of revolutions during residence time of fillers and the length of fibred fillers is very important factor affecting on the electric conductivity, so that it should pay attention to breakage of filler in case of fragile fibred filler.

As the kneading conditions, lower revolution speed and higher feeding rate were suggested.

The homogeneity of productions in continuous kneading improved with decrease of revolution speed because the lateral mixing was restrained.

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