Mixing Characteristics of an Internal Mixer: Uniformity of Mixed Rubber

Michiharu Toh,[1] Toyohiko Gondoh,[1] Tetsuo Mori,[1] Mamoru Mishima[2]

[1]*Department of Biochemistry and Applied Chemistry, Kurume National College of Technology, Komorino 1-1-1, Kurume City, 830-8555, Japan*
[2]*Sumitomo Heavy Industries, Mechanical and Equipment Ltd., 1501 Imazaike, Toyo City, 799-1393, Japan*

ABSTRACT: Uniformity of mixed rubber in an internal mixer equipped with three long-type rotor sets was investigated. After mixing butadiene rubber with ZnO powder for a certain period of time, a small amount of the mixed rubber was taken out from two points in the mixer: the center point of the reservoir and the side point of the sickle-shaped zone on the rotors. The concentration of ZnO in the sampled rubber was measured by the atomic absorption analyzer to know the dispersion of ZnO into the rubber. The twist direction and helix angle of rotors, and the position of ZnO powder incorporation, affected the uniformity of mixed rubber. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 95: 166–172, 2005

Key words: polybutadiene; rubber; mixing; dispersions twist direction

INTRODUCTION

In internal mixers, the mixing behavior of rubber with additives changes momentarily by a large helix angle, a large length/diameter ratio of rotor, a different rotation speed of two rotors, an existence of voids, etc. Freakley investigated the mixing characteristics of internal mixers quantitatively.[1]

We studied the mixing characteristics of an internal mixer equipped with four kinds of short rotors sets from the standpoints of the degree of mixing used for the evaluation of the mixing characteristics of powder mixers by Toh et al.[2,3] Four kinds of the (g)- and the (h)-type short rotors having helix angles of 5° and 15° were used. When the (g)-type rotors, having a concave rear part, were rotated under the equal rotor speed and phase angle of 90° in a two-dimensional internal mixer [L(length of rotor)/D(inside diameter of mixing chamber) = 0.20], rubber was well interchanged between the two lobes of the mixing chamber.[4] The (h)-type rotor is a conventional one, which has a symmetrical cross section of rotor. These rotors are short rotors, having L/D = 0.65. The degree of mixing of Teflon particles or ZnO powder into butadiene rubber (BR) in an internal mixer equipped with these rotors was studied by the following two methods: (1) counting the number of Teflon particles distributed in the mixed rubber and (2) a small amount of the mixed rubber was taken out from the same mixture batch at varying mixing times, followed by measuring the concentration of ZnO in the sampled rubber by the atomic absorption analysis. It was found that the (g)-type rotor with a helix angle of 15° was the best to attain the homogeneous mixing in the mixer. In these experiments, four sets of rotors have the same twist direction to give circular movement of the mixed rubber in the mixer.

However, when both the L/D ratio and helix angle of (g)-type rotors with same twist direction are large, the relative position of the two rotors changes appreciably with the longitudinal direction.

In this article, we investigated the effect of twist direction of rotors on mixing characteristics in an internal mixer equipped with three kinds of (g)-type long rotors sets.

EXPERIMENTAL

The experimental apparatus and procedures used were almost the same as described in the previous articles.[2,3]

Figure 1 shows the internal mixer used in this experiment, equipped with (g)-type long rotor sets hav-
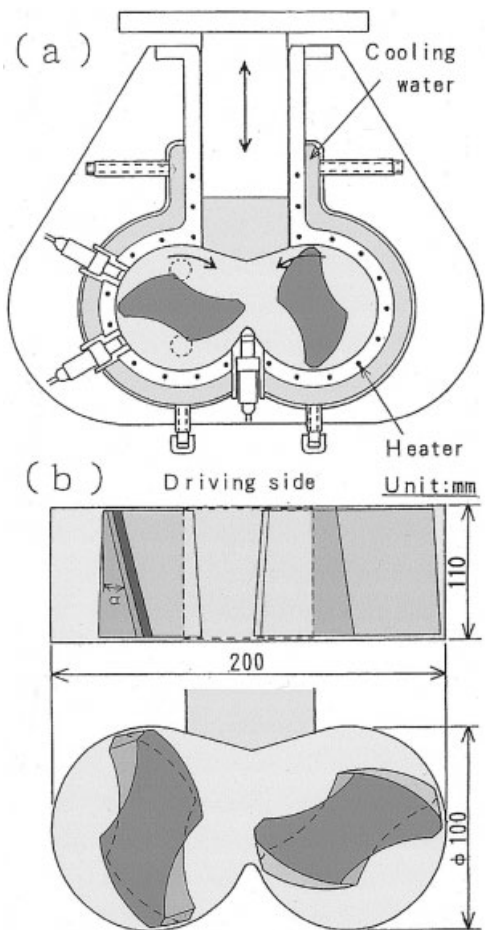
Figure 1 Designs of an internal mixer and rotors.

Figure 2 (a) Same twist direction of two (g) type rotors; (b) different twist direction of two (g) type rotors.

ing L/D = 1.10. Figure 2(a,b) shows the same and different twist directions of two rotors, respectively.

Experiments were carried out in the following method. BR used is JSRBR01. The ZnO powder was added in a ratio of 7.4 mg powder/g rubber. The fill factor ε was 0.8. That is, 6.77 g ZnO powder was wrapped in 6 g rubber. BR (909 g) was initially kneaded for a certain period. When the temperature of the rubber reached 100°C, the mixing was stopped. Then, the lump of the rubber including the ZnO powder prepared as mentioned above was incorporated below the ram at A or B position in Figure 3. The mixing time (T_M) was 1–60 min. The rotation speed (N_m) was 10 rpm. The rotors were always stopped at the same position at varying mixing times, and then a small amount of the mixed rubber was sampled in the center of the reservoir below the ram and above the bridge, and in the side of the sickle-shaped zone in front of the tip of the rotor in the mixer in Figure 3 at varying mixing times. The sampling time was $\sim$ 60–90 s.

The length, the diameter, and the weight of the sampled rubber were about 110 mm, ϕ 5 mm, and 1.0 g, respectively. The sampled rubber was cut into
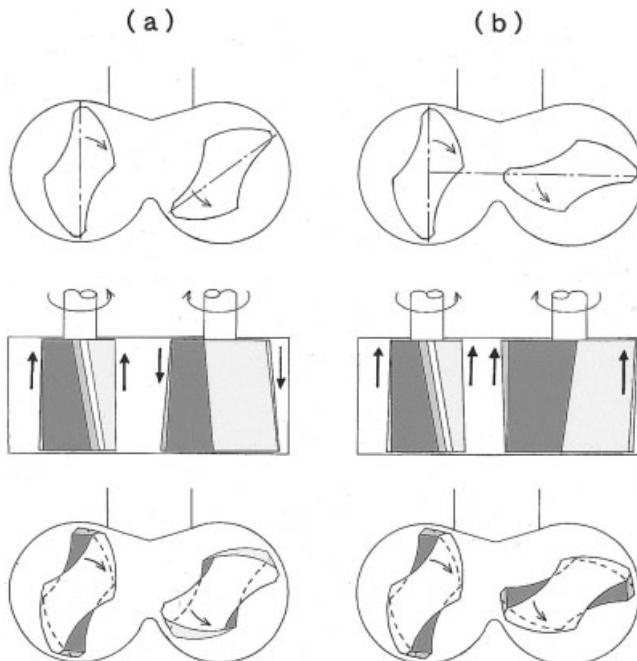
10 portions along the rotor axis as shown in Figure 3. The weight of these small rubber samples was $\sim$0.10 g. These rubber samples were burned for 1 h at 800°C in an electric furnace, and only ZnO was obtained.

ZnO was dissolved in HNO_3. The concentration of ZnO in the rubber was calculated from the concentration of Zn^{2+} in the solution measured by the atomic absorption analyzer (Shimazu AA-670G).

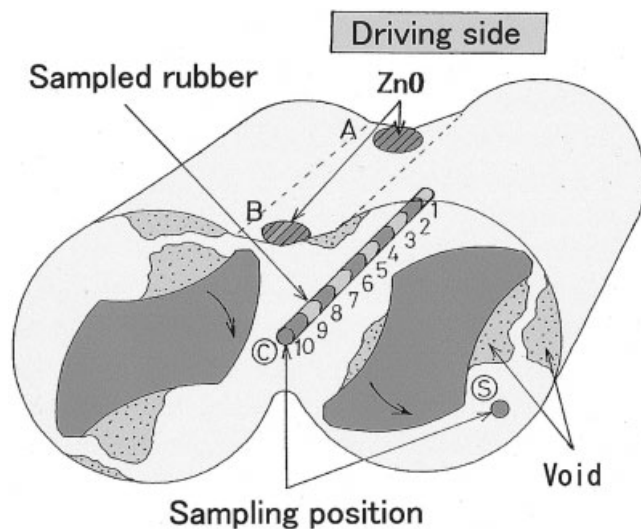C_0 is the concentration of ZnO per 1 g rubber at the start of mixing, and $\bar{C}$ is the average concentra-

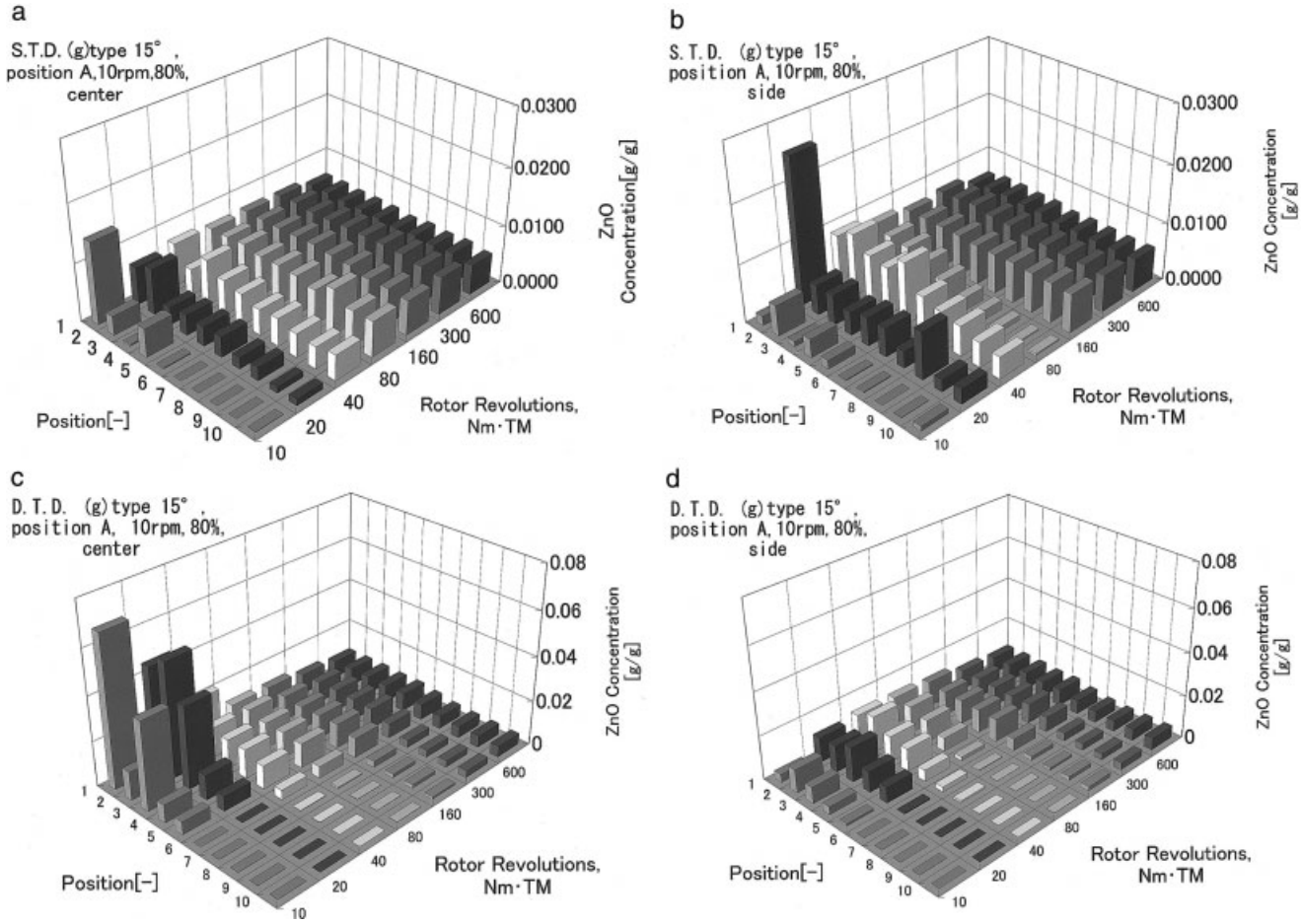**Figure 3** Schematic diagram of sampling positions and sampled rubbers.

Figure 4 Three-dimensional distributions of the concentration of ZnO powder. (a,c) Same twist direction of two rotors, (b,d) different twist direction of two rotors.

tion of ZnO per 1 g rubber. C_i is the concentration of ZnO in each small sampled rubber ($i = 1–10$) in the center of the reservoir and the side of the sickle-shaped zone. N is the number of the small sample rubber ($N = 20$).

The distribution of ZnO was analyzed by the degree of mixing. The degree of mixing (M) was obtained by the standard deviations of concentration of ZnO at the start of mixing σ_0 and that of each mixing time σ,

$$M = \sigma / \sigma_0 \tag{1}$$

$$\sigma_0 = \{C_0(1 - C_0)\}^{0.5} \tag{2}$$

$$\sigma = [\{\Sigma(C_j - C)^2/(N - 1)\}]^{0.5} \tag{3}$$

where

$$C = (\Sigma C_j)/N \quad (j = 1–20)$$

RESULTS AND DISCUSSION

Figures 4, 5, and 6 show the three-dimensional distributions of the concentration of ZnO, the variation of M with rotor revolutions (NmT_m), and the two-dimensional distributions of the concentration of ZnO at $NmT_M = 600$, respectively, for (g)-type rotors having helix angle $\alpha = 15°$ at the fill factor $\varepsilon = 0.80$ and the rotating speed $Nm = 10$. Figures 4(a), 5(a), and 6(a) show those for the same twist direction of two rotors and Figures 4(b), 5(b), and 6(b) show those for the different twist directions of the two rotors. Comparing Figure 5(a, b), the degree of mixing (M) achieved for the same twist direction is smaller than that for the different twist directions at $NmT_M = 600$. It is considered that rubber was slowly mixed by a folding process at a low rotating speed of this type mixer, and this process was different from subdivision-intercorporation process of high rotating speed of Banbary-type mixer. So, there will be fluctuations of M at the low rotor revolutions, as shown in Figure 5(a). M decreased gradually with high rotor revolutions and the uniformity of the mixed rubber was attained. In addi-
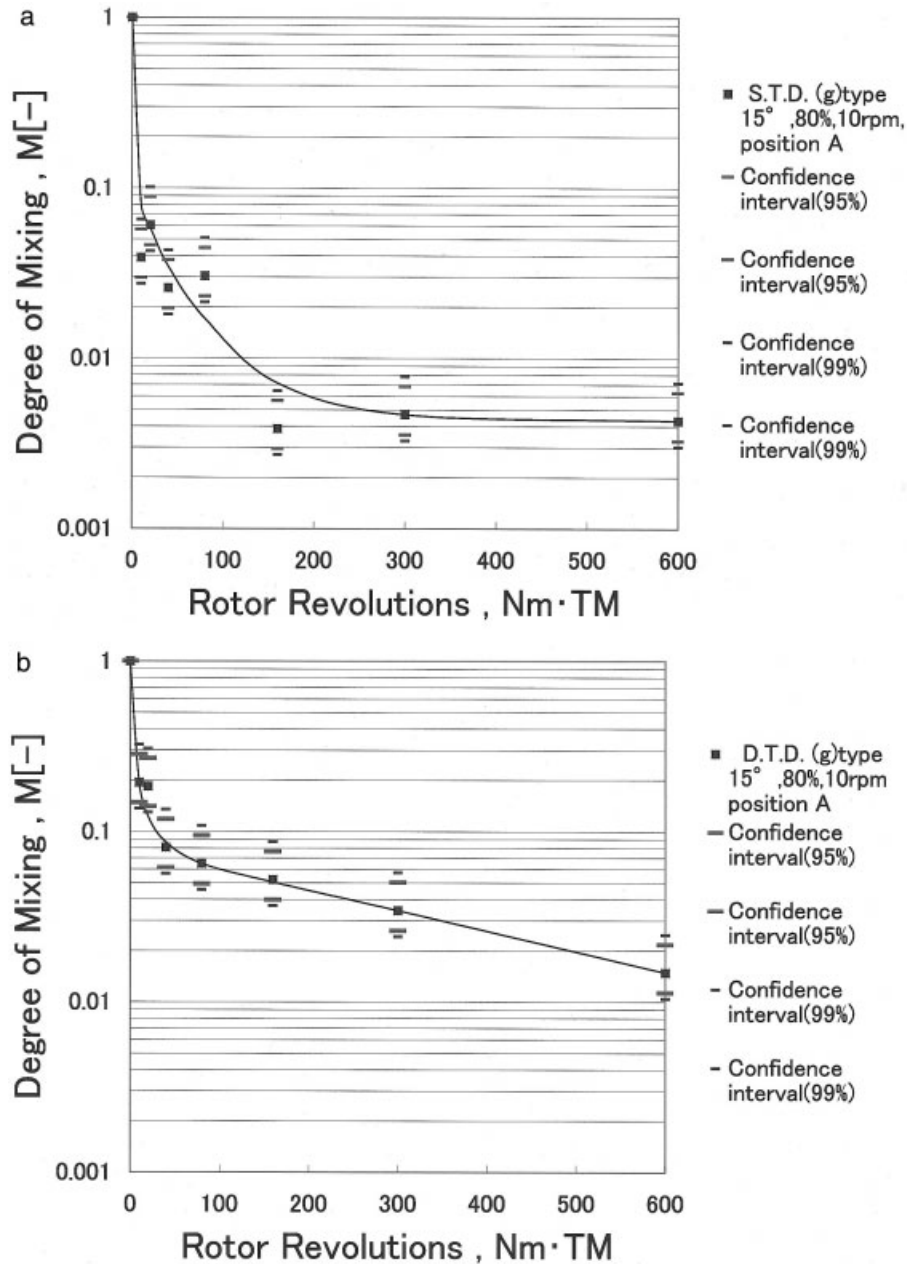
Figure 5 Variation of M with NmT_M. (a) Same twist direction of two rotors, (b) different twist direction of two rotors.

tion, the uniformity of the mixed rubber increased by circular movement of the rubber in the mixer as shown in Figure 2(a). The concentration of ZnO for the same twist direction is almost constant at all positions, but is smaller than that at the start of mixing, as shown in Figure 6(a). On the other hand, the concentration of ZnO for the different twist directions varies longitudinally, as shown in Figure 6(b). Also, the concentration of ZnO at the longitudinal position in the center of the reservoir is almost equal to that of the side of the sickle-shaped zone. This suggests that rubber was well interchanged and mixed at vertical cross sections to rotor axis, although the longitudi-

nal movement of rubber is poor. High values of M in Figure 5(b) and the concentration distribution of ZnO in Figure 6(b) show that the uniformity of the mixed rubber was not attained.

In the above-mentioned experiments shown in Figure 4, 5, and 6, ZnO powder was incorporated below the ram at position A in Figure 3. In the following experiments, ZnO powder was incorporated below the ram at position B in Figure 3.

Figures 7 and 8 show the variation of M with NmT_M and the two-dimensional distributions of the concentration of ZnO at $NmT_M = 600$, respectively. The (g)-type rotor having helix angle $\alpha = 15°$ and
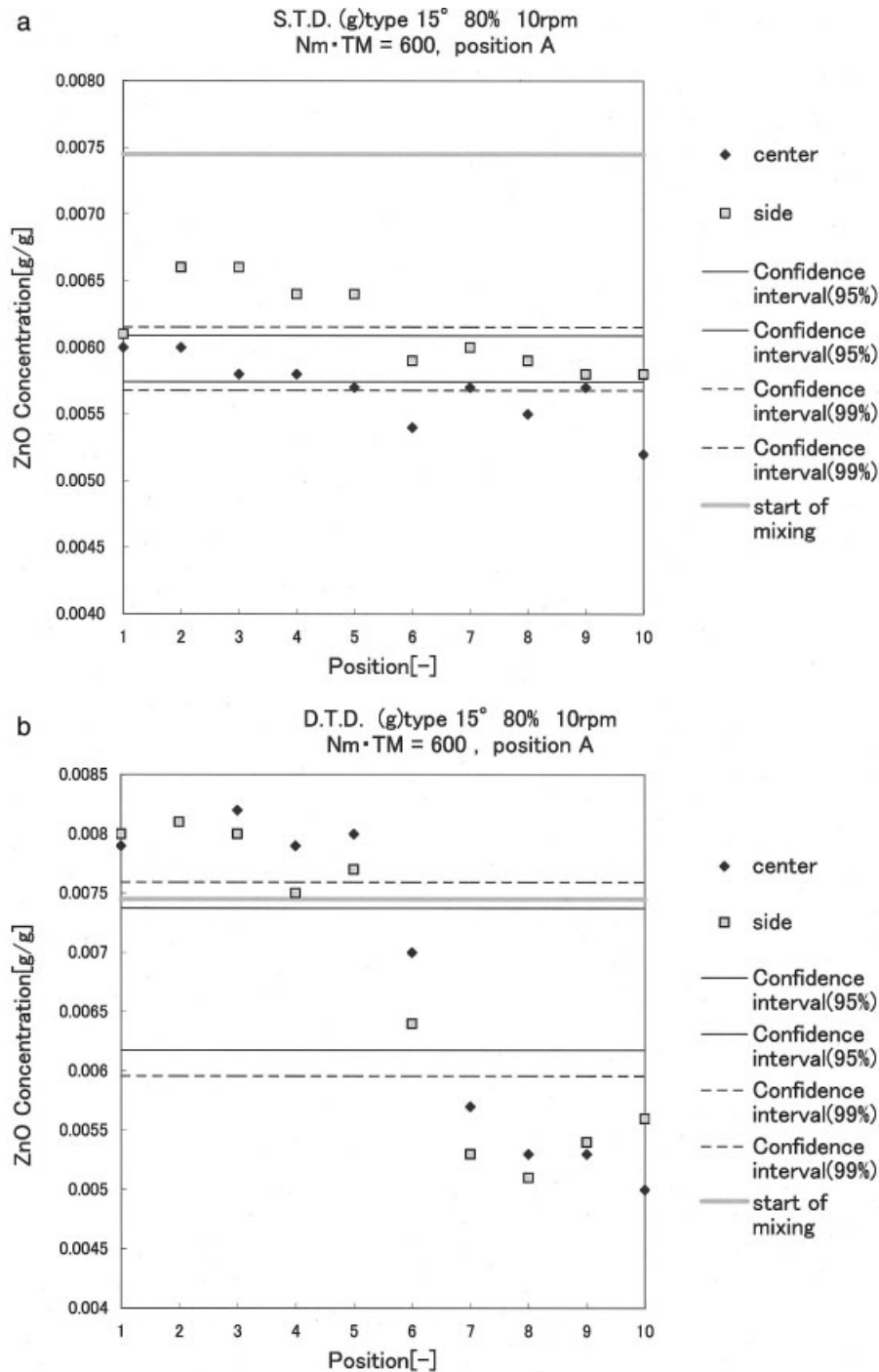
Figure 6 Two-dimensional distributions of the concentration of ZnO powder at $NmT_M = 600$, (a) same twist direction of two rotors, (b) different twist direction of two rotors.

the same twist direction were used at $\varepsilon = 0.80$ and $Nm = 10$.

Comparing Figure 5 with Figures 7 and 8, a good degree of mixing and uniformity was achieved when ZnO was incorporated at position B. This suggests that the relative position of two rotors below position B was effective for mixing.

Figures 9 and 10 show the results for the (g)-type rotor having a helix angle of $\alpha = 5°$ and the same twist

direction. The value of M fluctuates under the low rotor revolutions, as shown in Figures 7 and 9. This suggests that rubber was mixed slowly by folding process at low-rotating speed of the mixer. Comparing Figures 5, 7, and 8 with Figures 9 and 10, the excellent degree of mixing was achieved for the rotor having a helix angle of $\alpha = 5°$. This suggests that the helix angle is small and the relative position of two rotors does not change appreciably along the longitudinal direc-

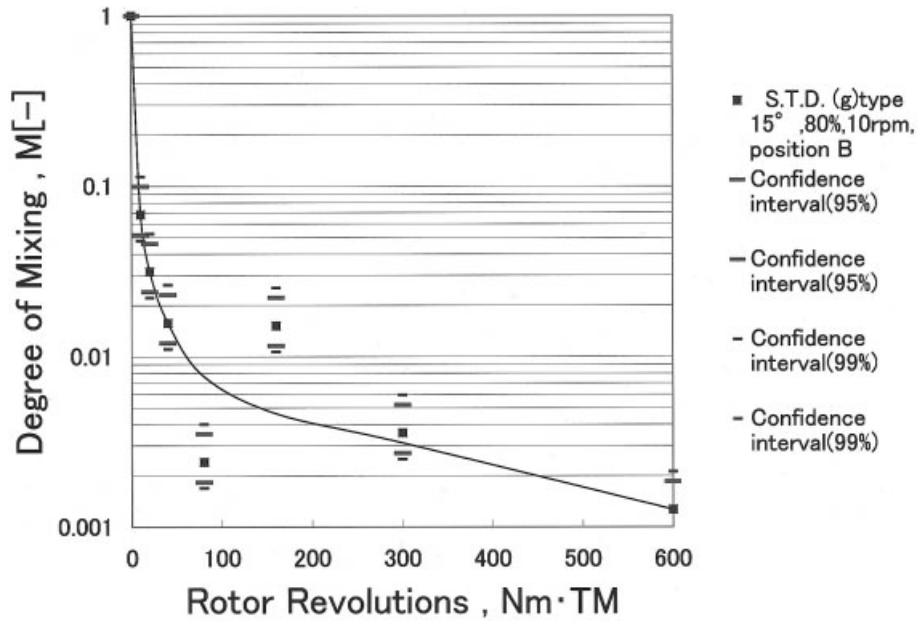**Figure 7** Variation of M with NmT_M for same twist direction of two rotors.

tion; therefore, the good combination of interchange of rubber between two lobes and longitudinal circulation of rubber may be obtained.

CONCLUSION

ZnO powder was mixed with BR rubber in an internal mixer with three kinds of (g)-type long rotors sets. The variation of the degree of mixing with NmT_M was almost continuously measured to determine the ZnO concentration. A small amount of the mixed rubber was taken out at various mixing times from two sampling positions of the reservior and the sickle-shaped zone in front of the tip of the rotor in the internal mixer.

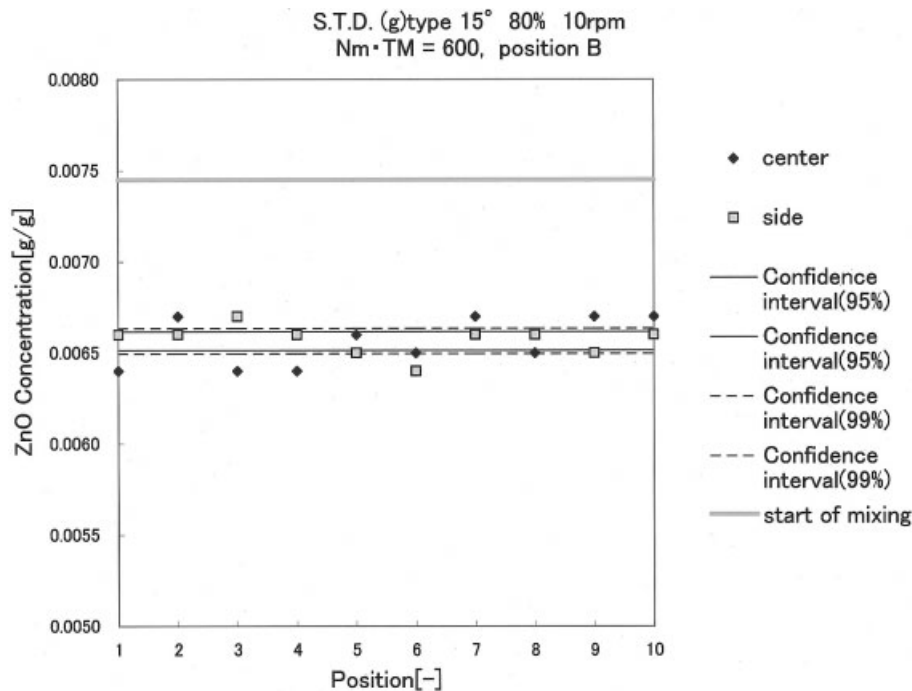It was found that when both L/D ratio and helix angle of (g)-type rotors with same twist direction were

Figure 8 Two-dimensional distributions of the concentration of ZnO powder at $NmT_M = 600$, for same twist direction of two rotors.
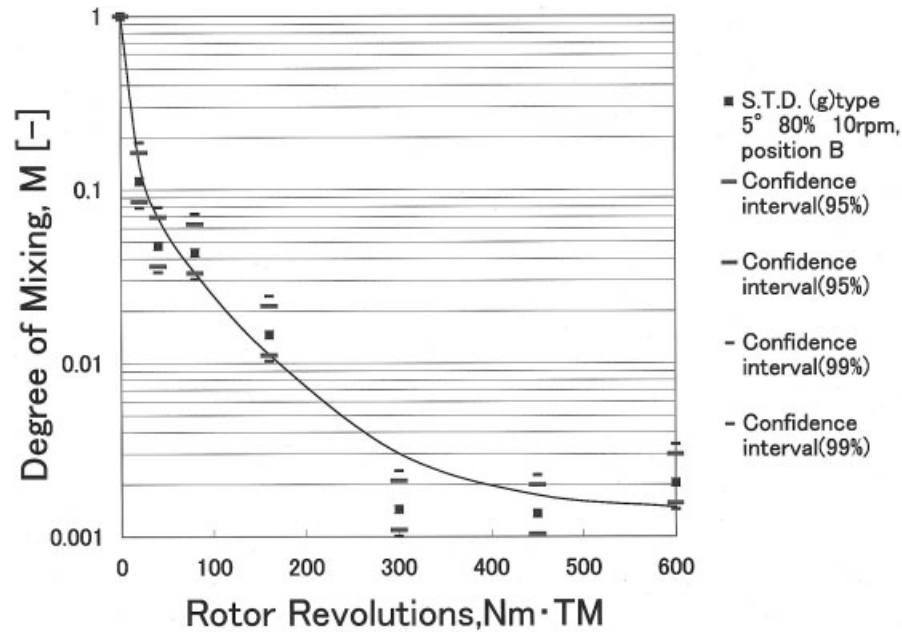
Figure 9   Variation of M with NmT_M for same twist direction of two rotors.
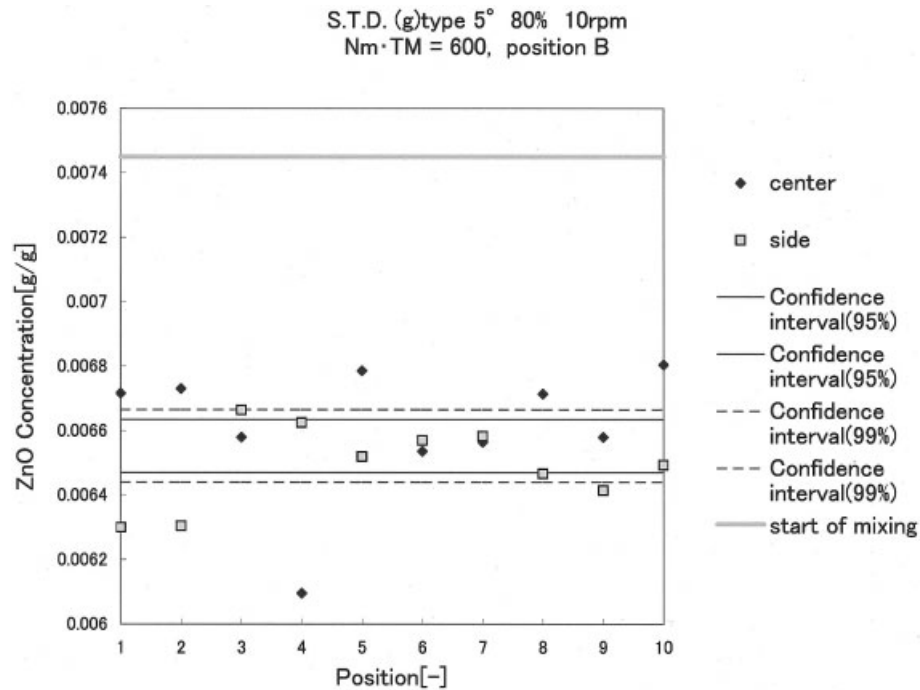

Figure 10   Two-dimensions of the concentration of ZnO powder at NmT_M = 600, for same twist direction of two rotors.

large, the relative position of the two rotors changed appreciably with the longitudinal direction. The same twist direction of the (g)-type rotor having a nonsymmetrical cross section had a good degree of mixing, when the ZnO powder was incorporated at appropriate positions and the relative position of the two rotors was effective for the interchange of rubber between two lobes and longitudinal circulation of rubber.

References

1. Freakley, P. K. Rubber Chem Technol 1992, 65, 706.
2. Toh, M.; Gondoh, T.; Mori, T.; Murakami, Y.; Nishimi, H.; Mishima, M. Rubber World 1997, 215 (6), 29.
3. Toh, M.; Gondoh, T.; Mori, T.; Murakami, Y., Satoh, H.; Kuratsu, M.; Mishima, M. Full Texts of IRC97(Malaysia) 1997, 1041.
4. Toh, M.; Gondoh, T.; Mori, T.; Hara, S.; Murakami, H. J Appl Polymer Sci, Appl Polym Symp 1992, 50, 133.