Scale-up Rule of Double Bubble Tubular Film Production Process for Nylon6 Film

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ABSTRACT

The analysis of scale-up rule for double bubble tubular film process of nylon6 was investigated. The applicability of the scale-up rule which was set up theoretically was evaluated by using both the small scale research machine and the large scale production machine under the conditions of controlling the relationship between output and film width etc. according to the scale - up rule, namely bubble radius R, film thickness H and output rate Q for the small scale test machine and KR, LH and K^2LQ for the large scale production machine respectively.

Both small scale test machine and large scale production one showed the same stretching stability, the equivalent stretching stress and the equivalent birefringence pattern. In the observation through the polarizing plate of the bubble sample which shows deformation pattern, the equality of deformation pattern was also confirmed.

It is found that the scale-up rule which was set up theoretically is applicable to predict the physical properties and bubble stability for large scale double bubble tubular film process once the experiment is carried out by using the small scale machine and a small amount of resin.

1. Introduction

In recent years, the environmental problem has come into question in the packaging industry. As these problems, the dichlorination and the refuse reduction have been closed up in this industry. Especially this refuse reduction has been a serious problem, so it is desired that this problem will be solved. In order to achieve this refuse reduction, there is a rapid increasing shift from a bottle to a standing pouch for repackaging use in an effort to utilize resources effectively.
For the provision of this standing repackage pouch, thin and strong biaxial oriented nylon film is indispensable.

As shown above, the demand for the biaxially oriented nylon film has become popular. Several kinds of manufacturing processes have been developed to produce this biaxially oriented nylon6 film featured by high strength. Among them the double bubble tubular process producing biaxial stretched nylon film is the best one in terms of impact strength.
It is valued highly for the purpose of distribution safety of products.

The technology which was established by the small scale research machine must be applied to the large scale production one. It was examined for the purpose of the demonstration of scale-up theory of the double bubble tubular stretching technology. This double bubble tubular film process is closely related to the tubular film one, so we use the basic studies reported on the tubular film process.

Studies on the tubular film process were investigated by various researchers.
The earliest investigations were published by Alfrey [1] and more specifically by Pearson[2] generally considering kinematics and stress-analysis by membrane theory. In a subsequent series of papers, Pearson and Petrie elaborated on this analysis and made specific calculations for an isothermal Newtonian fluid model. An isothermal viscoelastic model was described by Petrie[3]. Analysis of temperature fields and their interaction with kinematics was first considered in papers by Han and Park[4] and Petrie in 1975. These efforts were continued by Wagner [5-6] in a later paper.

Kanai and White [7] considered local kinematics and heat transfer rates as well as bubble stability.

Those results were used as the basis for construction of a model of the dynamics, heat transfer and structure development in tubular film extrusion. A later reports [8] represented an advance on earlier papers on modeling by inclusion of crystallization and more quantitative representations of local heat transfer rates. These research works apply for theoretical analysis of tubular film extrusion and its application for HMW-HDPE [9] . Further using the theoretical equations on tubular film extrusion, the authors presented a scale-up rule [10].

There are several reports issued on double bubble tubular stretching technology [11-22]. The analyses of deformation behavior of nylon 6 were reported previously according to stretching stress analyses [23]. The previous paper discussed three topics: relationship between process condition and film stretching stress; deformation rate during double bubble process, birefringence; and scale-up rule for double bubble tubular film process.
In this time two types of machines which are small scale research machine and large scale production one are used. The stretching bubble stop samples were evaluated made by small scale research machine and large scale production one.

2. Experimental

2.1 Experimental Equipment

The apparatus of the double bubble tubular film process shown in Figure 1 was used.
By using an extruder (L/D=24) with the diameter of 40mm as well as a circular die with the diameter of 75mm and the lip clearance of 1mm, non-stretched film for small scale research machine was produced at the condition of the resin temperature 265℃, blow-up ratio of 1.2, with a water-cooling ring having the diameter of 90mm for small scale research machine.

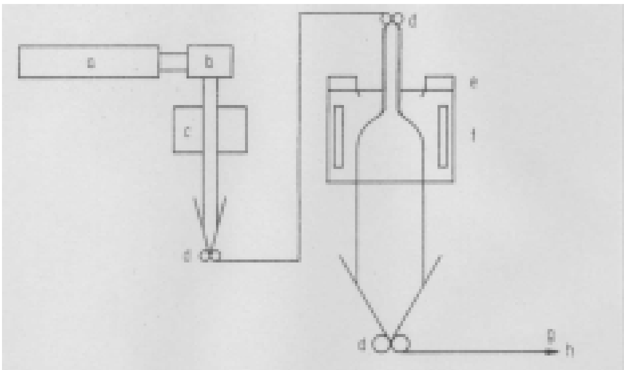


Fig. 1. Schematic view of double bubble tubular film process
a: extruder, b: die, c: cooling bath, d: take up roll, e: air ring, f: heating, g: annealing, h: winding

By using an extruder (L/D=25) with the diameter of 115mm as well as a circular die with the diameter 300mm and lip clearance of 1 mm, non-stretched film for large scale production machine was produced at the condition of the resin temperature 265℃, blow-up ratio of 1.1, and with a water-cooling ring having the diameter of 330mm.

This non-stretched film is stretched simultaneously in the machine direction and transverse one by using inside bubble air, a drawing machine composed of two pairs of pinch rolls and a heating furnace (a far infrared radiation heater is self-contained).

The stretched film is heat-set using tentering process with heat treatment device.

2.2 Material

The material is Ube Nylon 1024FDX14 (Nylon 6) with the relative viscosity of η_r=3.75 (sulfuric acid concentration 98%) and mean molecular weight of 24000.

2.3 Experimental Method

The formation of the scale up rule is confirmed and is evaluated experimentally using small scale research machine and large scale production one of the tubular biaxial stretching equipment.

The process conditions of non-stretched film are 265℃ for resin temperature at the die exit, 1.2 for blow up ratio, and 6.0 for draw down ratio respectively, and a water cooling method was used to lower crystallinity. The cooling water temperature was adjusted to be 18℃.

The stretching process consists of a heating/ stretching furnace and an air ring device. The air ring device was installed at the upper part of the heating furnace so as to fix the stretching start point, and it injected air downward at the angle of 45 ° .

The standard condition for stretching process was set at 310^0C for process temperature (heater set temperature) and MD (Machine Direction)/ TD (Transverse Direction) =3.0 /3.2 for stretching ratio respectively.

The film thickness is 130 μm for non-stretched film, and it became 13.5 μm after stretching. The stretched film was heat treated, using a thermosetting device of the tentering process to prevent shrinkage, thereby there is no disturbance due to film shrinkage permitting the measurement of physical properties alike.

The samples were collected under 2 conditions in which the deformation rates are different.

1.0 and 0.6 were selected as deformation rate. Film was made under 2 conditions by small scale research machine and large scale production machine. (Table 2)

2.4 Evaluation of the stretching stress and bubble stability

The bubble internal pressure was obtained using the digital manometer (Yokogawa Hokushin). The stretching stress was calculated by the measurement of bubble inside pressure of the stretching developing.

The bubble diameter change was measured in order to evaluate the stability of the stretching bubble.

2.5 Shape Comparative Evaluation of the stretching deformation bubble

The bubble stability is closely related to the deformation film, but it is very difficult to measure the deformation pattern during the second bubble inflation, because the heating apparatus covers bubble.

In order to analyze the behavior of deformation pattern, the extruder and take-up device were suddenly stopped and bubble sample was collected. Bubble width pattern and film thickness pattern at each position of the bubble sample were measured by using stopped bubble sample. Stretching ratio at each position was calculated by using bubble width pattern and film thickness one.

2.6 Evaluation of Birefringence

The Birefringence evaluation of the stretched film sample produced by small scale research machine and large scale production one was carried out.

The birefringence was measured by using BH2 (type Olympus BH-2, Japan) at each point.

2.7 Observation of Deformed Bubble through Polarizing Plate

The deformed samples under the various conditions during the stretching process were taken and each of them was inserted between two polarizing plates crossed with each other to observe and assess the deformation state of stretching bubbles by shedding light from below. Each sample under each condition was photographed using a high sensitivity film.

The anisotropy of the orientation of film from the stretching start point to stretching end point was observed. The observation evaluation of stretching bubble stop sample of small scale research machine and large scale production one was carried.

2.8 Evaluation of the Crystal Form

Crystal form evaluation of the stretch film sample produced by small scale research machine and large scale production one was carried out. The way of the change of the crystal form of stretching deformation process of nylon film was evaluated using the infrared absorption evaluation(Infrared spectroscopy).

The value of the α type crystal form used 977 cm^{-1} absorbency, and the value of γ type crystal form using 928 cm^{-1} absorbency. The ratio of α type crystal and γ type crystal was measured and was evaluated continuously for the flowing direction of the bubble.
The ratio α and γ was expressed α/γ.

2.9 Evaluation of the Film physical property

Physical property evaluation of the stretch film sample got by small scale research machine and large scale production one was carried out. Film impact strength, penetrate strength, tensile strength at break and tensile elongation at break are measured.

3. Theoretical background

The stretching ratio of both MD and TD are determined by the inside bubble pressure and the different roll speeds between top rolls and bottom rolls. In this manner the second bubble is simultaneously stretched in both machine direction and transverse direction.
Fig.2 shows the measurement method of stretching stress. Stretching stresses were calculated with help of Eqs.1 and 2,

$$\sigma_{MD} = (\Delta P \cdot R)/H_L \qquad (1)$$
$$\sigma_{TD} = (T/r)/2\pi H_L R \qquad (2)$$

Where ΔP, T, R, r, and H_L are inside bubble pressure, torque, final bubble radius, radius of bottom nip roll and final film thickness respectively.
The calculation of deformation rate D_f is defined as follows.

$$D_f = D_0/D \times 1/t \times 1/10 \qquad (3)$$

Where D_0, D and t are thickness of non-stretching film, thickness of stretching film and time of deformation respectively.

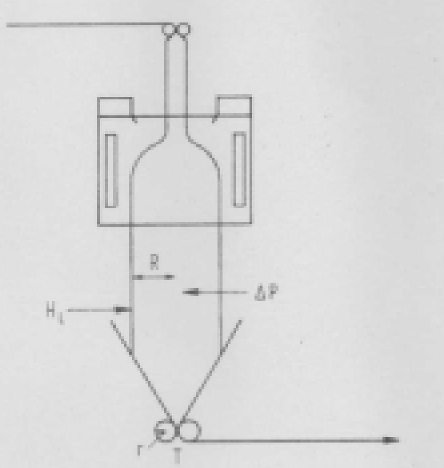


Fig. 2. Measurement method of stretching stress

The force balance on the double bubble tubular film is developed from membrane theory. The membrane theory leads to a balance of forces on the film between positions Z and take-up position L. This has the form:

$$F_L = 2\pi RH\sigma_{MD}\cos\theta + \pi\ (R_L{}^2 - R^2) \cdot \Delta P \qquad (4)$$

The stress σ_{MD} and σ_{TD} are related to the pressure ΔP through the expression:

$$H \cdot \sigma_{MD} / R_1 + H \cdot \sigma_{TD} / R_2 = \Delta P \qquad (5)$$

Where F_L is the bubble tension, R_L the final bubble radius, and R_1 and R_2 are appropriate radii of curvature.

$$R_1 = ((1 + dR/dZ)^2)^{3/2} / d^2R / dZ^2 \cdot R_2 = R / \cos\theta \qquad (6)$$

The maximum stretching stress σ_{MD} and σ_{TD} are closely related to the physical properties of film. The maximum stress at the stretching final is used to set up the scale-up rule.
At the stretching final point, bubble diameter is equal to final bubble diameter.

$$\sigma_{MD} = F_L / 2\pi R_L H_L \qquad (7)$$

$$\sigma_{TD} = \Delta P \cdot R_L / H_L \qquad (8)$$

H_L: final film thickness.
By using the analysis of dimensionless terms, stress σ_{MD} and σ_{TD} are presented by following equations.

$$\sigma_{MD} = (A+B\ (R_L/R_0)^2) / 2\pi \cdot Q\eta_0 = G_1 / Q\eta_0 / R_0 R_L H_L \qquad (9)$$

$$\sigma_{TD} = B/\pi \cdot Q\eta R_L / R_0{}^3 H_L = G_2 \cdot Q\eta R_L \cdot / R_0{}^3 H_L \qquad (10)$$

Q: out-put rate , η_0: viscosity , R_0: initial bubble radius, Z_L: axial distance between beginning point and final point of bubble inflation where A and B are constants under the same conditions of R_L/R_0, V_L/V_0, and Z_L/R_0, and G_1 and G_2 are constants under the condition of constant stretching ratio of machine direction, stretching ratio of transverse direction.
It is found that stretching stress was a function of film thickness and square of bubble diameter. (Table 1) In case of changing bubble radius R and film thickness H into KR and LH, output rate K^2LQ is needed.

4. Results and Discussion

4.1 Evaluation of the stretching stress

The stretching stress level by small scale research machine and large scale production one was identical, and the formation of the scale-up rule was confirmed. (Table 3)
The uniformity of the bubble diameter and the bubble stability were also reproduced.
When the deformation rate was decreased, the stretching stress lowered and the bubble stability also lowered.

4.2 Comparative Evaluation of the stretching deformation and bubble shape

Figure 3 shows typical example of bubble shape during the second bubble inflation.
In Figure 4 and 5, the stretching ratio of transverse direction increases slowly under the low deformation rate. The stretching ratio of machine direction increases rapidly compared with the one of the transverse direction.

The difference between stretching ratio in machine direction and transverse direction were calculated. As shown in Figure 6 and 7, the stretching ratio of machine direction is larger than the one of transverse direction at all position. Especially in the condition of low deformation rate, the stretching ratio of machine direction has larger value than in the transverse direction.

It was confirmed that the stretching deformation condition agreed with the measuring result of small
scale research machine and large scale production one by adjusting the size scale.
This result suggests that the scale up rule of bubble diameter is applicable.

Fig. 4 Small scale (Deformation rate 1.0)

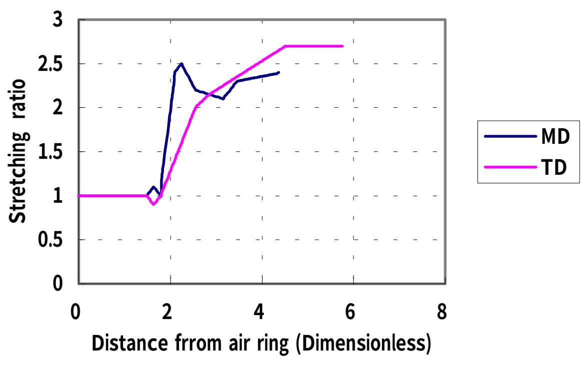


Fig.4. Small scale (Deformation rate 0.6)

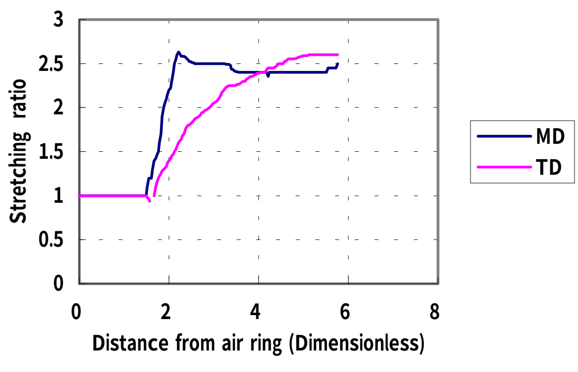


Fig.5. Large Scale (Deformation rate 1.0)

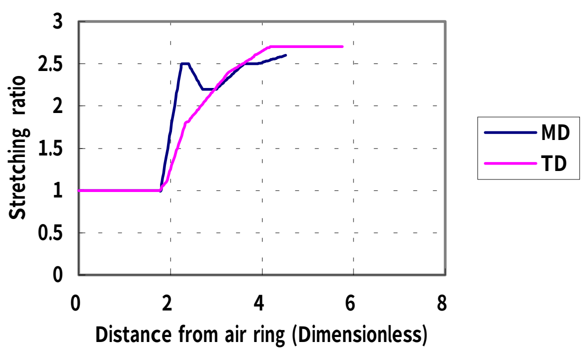
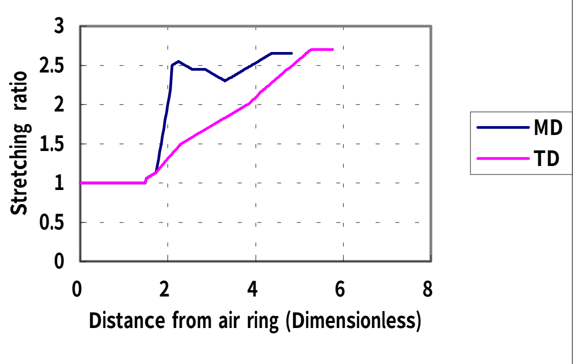
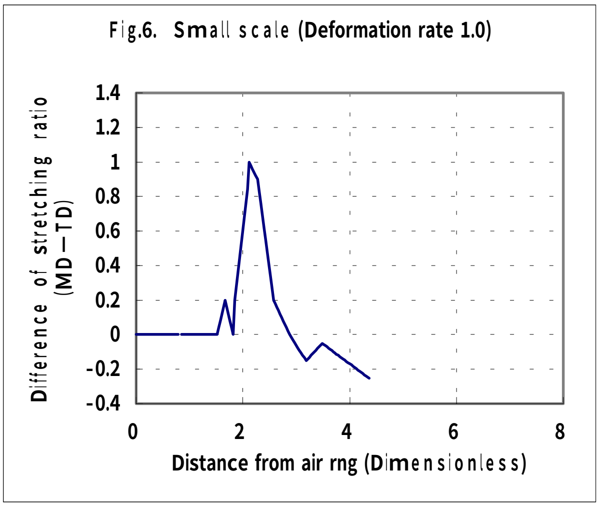
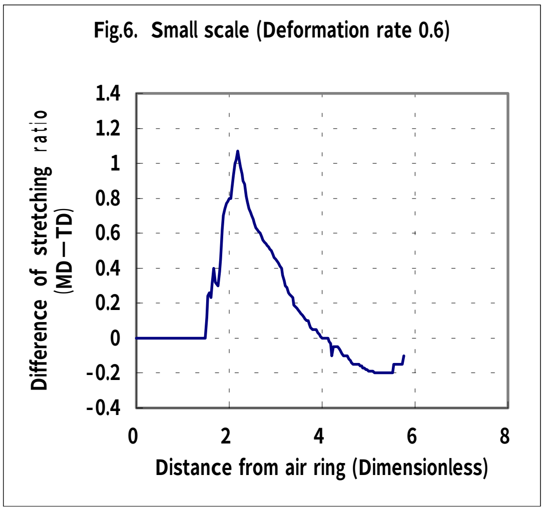
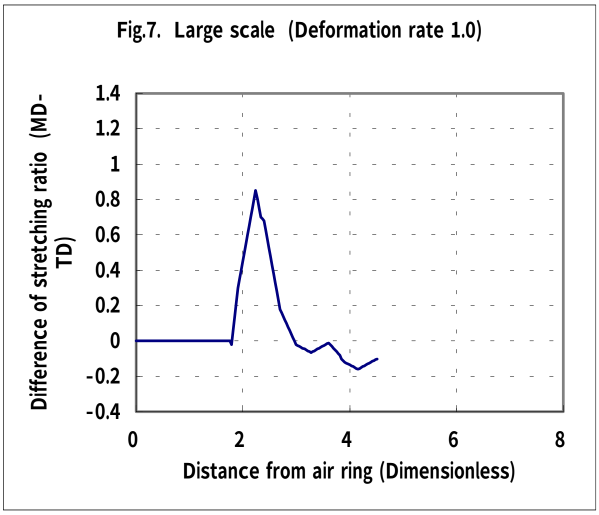


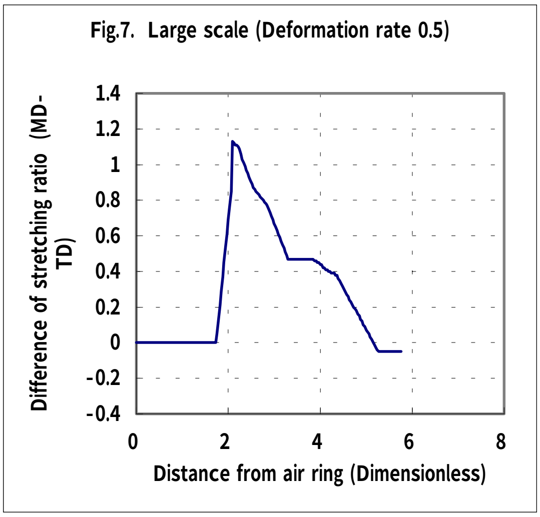
Fig.5. Large scale (Deformation rate 0.5)











4.3 Evaluation of the Birefringence

The birefringence of stop sample in each position was measured. Figure 8 and 9 show the relationship between birefringence and distance from air ring under the different deformation rates. Birefringence under the low deformation rate is clearly larger than one under the high deformation rate. These results correspond to the experimental ones obtained stretching ratio difference shown in Figure 6 and 7. As a result of evaluating the birefringence of the stretching developing sample of small scale research machine and large scale production one, it was found that birefringence pattern by the deformation process was almost identical.

Fig.8. Small scale (Deformation rate 1.0)

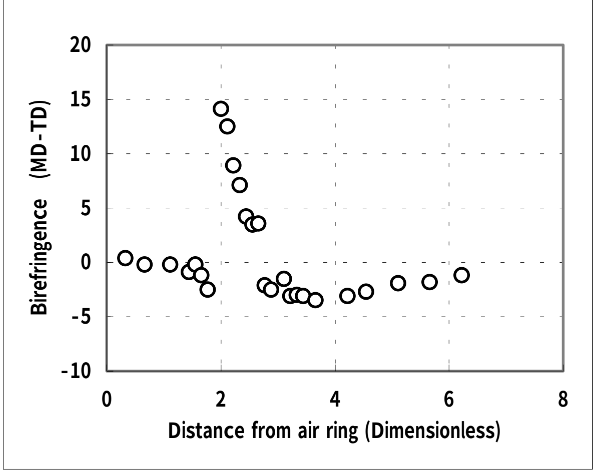


Fig.8. Small scale (Deformation rate 0.6)

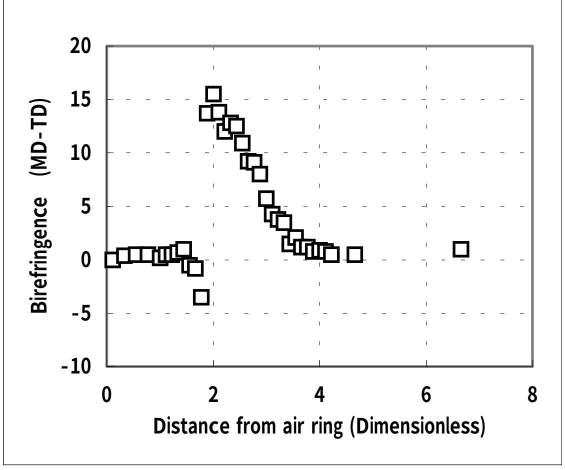


Fig.9. Largel Scale (Deformation rate 1.0)

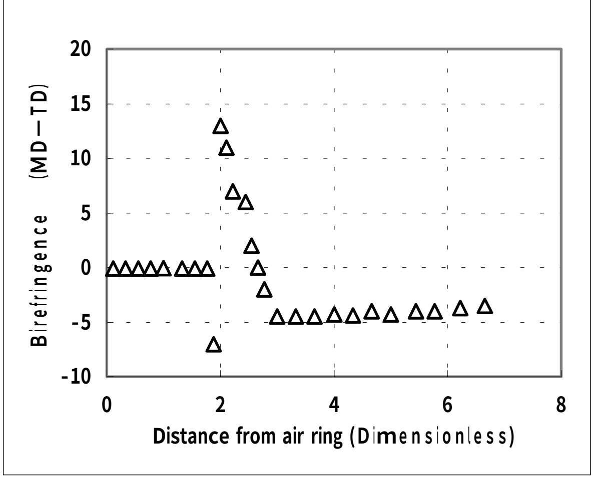
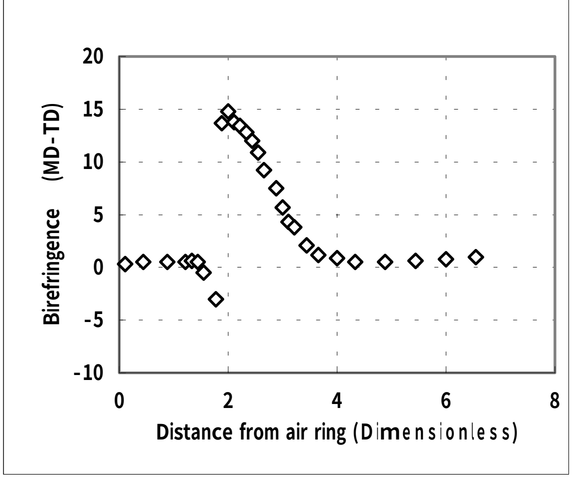


Fig.9. Large scale (Deformation rate 0.5)



4.4 Observation of Deformed Bubbles through Polarizing Plates

For the purpose of observing deformed bubbles stretching of film depending on stretching conditions, a suddenly stopped and cut sample during stretching process was taken under the specified process conditions, and a comparative evaluation was studied in terms of anisotropy through the polarizing plates.

Figure 10 and 11 show the results through the polarizing plate of the deformed bubble samples. It suggests that the deformation range is broad under the low deformation rate. This observation has close relation to the stretching ratio pattern. As a result of observation of deformed bubble sample of small scale research machine and large scale production one, it was found that deformation pattern by the deformation process was almost identical.

Fig.10 Observation of polarizing plate of second bubble sample
for small scale research machine

<Deformation rate 1.0 > <Deformation rate 0.6 >

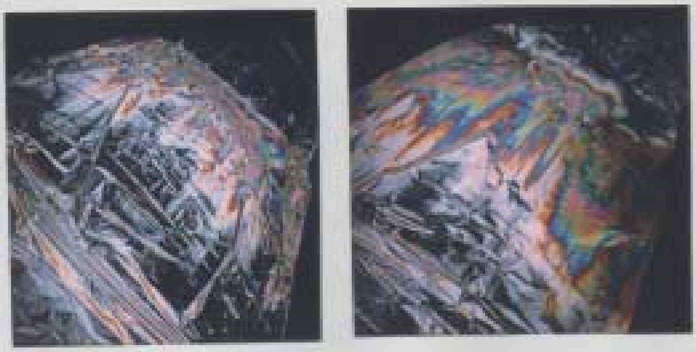


Fig.11 Observation of polarizing plate of second bubble sample
for large scale production machine

<Deformation rate 1.0>

4.5 Evaluation of the Crystal form

When the stretching condition was different, the way of the change of the crystal form of the deformation process differed. In Figure 12 and 13, the ratio of α to γ (α／γ)increases slowly under the low deformation rate. As a result of evaluation of the ratio α and γ (α／γ) of deformed bubble sample of small scale research machine and large scale production one, it was found that crystal form pattern by the deformation process was almost identical.

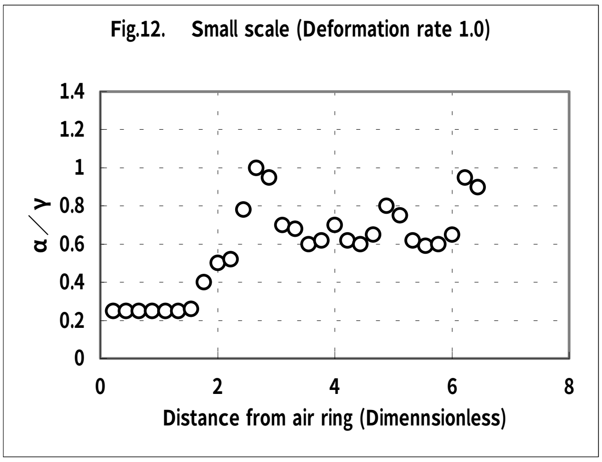


Fig.12. Small scale (Deformation rate 1.0)

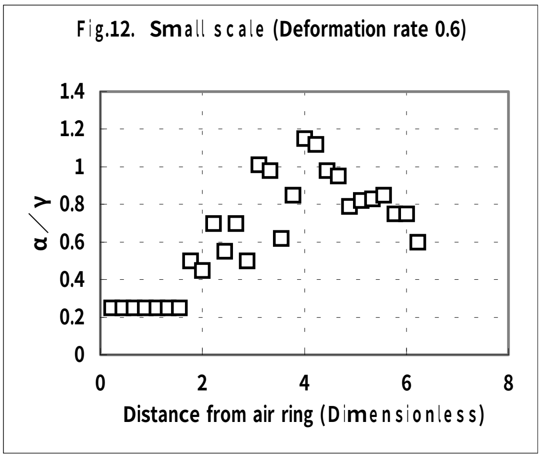


Fig.12. Small scale (Deformation rate 0.6)

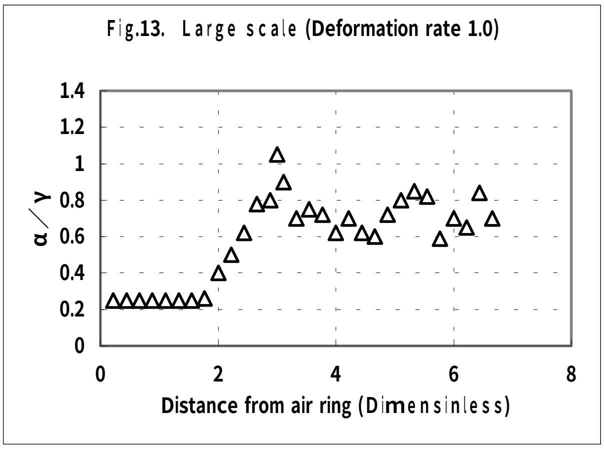


Fig.13. Large scale (Deformation rate 1.0)

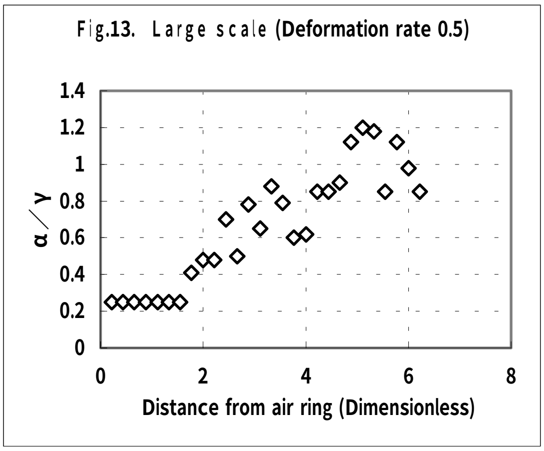


Fig.13. Large scale (Deformation rate 0.5)

4.6 Evaluation of the Film Physical Property

Film physical property has close relation to the stretching ratio pattern.
The scale-up aptitude of small scale research machine and large scale production one was evaluated using change process of the film physical property. Film impact strength, penetrate strength, tensile strength at break and tensile elongation at break were showed also same value. (Table 4) As a result of evaluation of film physical property of small scale research machine and large scale production one, it was found that film physical property by the deformation process was almost identical.

In case of changing bubble radius R and film thickness H into KR and LH, We can get same bubble stability, deformation style and film physical property to keep output rate K_2LQ. Namely the scale-up rule is applicable to keep the same deformation rate.

The scale-up rule is applicable to predict the physical properties and bubble stability.
As a result of the experimental evaluation, we found that the stretching stress and deformation pattern were very important factors to produce biaxially oriented nylon 6 film by double bubble

tubular process. We can predict the bubble stability and film physical properties for large scale double bubble tubular film extrusion is carried out by using the small scale research machine and a small amount of resin.

5. Conclusion

As a result of the study on the scale-up rule for the double bubble tubular process, the conclusion was obtained as follows.

(1) The application of the scale- up rule which was set up theoretically was evaluated by using both the small scale research machine and the large scale production machine under the conditions of controlling the relationship between output and film width etc. according to the scale-up rule, namely bubble radius R, film thickness H and output rate Q for the small scale test machine and KR、LH and K^2LQ for the large scale production machine respectively.

(2) Both small scale test machine and large scale production one showed the same stretching stress stability, the equivalent stretching stress , the equivalent birefringence pattern and the equivalent crystal form pattern, when we keep the same deformation rate and temperature.

(3) In the observation through the polarizing plate of the bubble sample which shows deformation pattern, the equality of deformation pattern was also confirmed.

(4) It is found that the scale-up rule which was set up theoretically is applicable to predict the physical properties and bubble stability for large scale double bubble tubular film process once the experiment is carried out by using the small scale machine and a small amount of resin.

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Table 1 Scale-up rule

Diameter	Thickness	Out put rate	σ_{MD}	σ_{TD}
R_0	H	Q	1	1
KR0	LH	Q	$1/K^2L$	$1/K^2L$
KR0	LH	K^2LQ	1	1

Table 2 Scale-up conditions
<Main condition>

Item	Small Scale Research Machine	Large Scale Production Machine	Magnification
Deformation rate	1.0	1.0	1.0
Tube Diameter	90 (mmφ)	330 (mmφ)	3.7
Output Rate	17.6 (Kg/Hr)	240.0 (Kg/Hr)	13.6
Thickness	130 (μm)	130 (μm)	1.0
Stretching Ratio	3.0／3.2	3.0／3.2	1.0

<Comparison condition>

Item	Small Scale Research Machine	Large Scale Production Machine	—
Deformation rate	0.6	0.5	—
Tube Diameter	90 (mmφ)	330 (mmφ)	—
Output	10.6 (Kg/Hr)	120.0 (Kg/Hr)	—
Thickness	130 (μm)	130 (μm)	—
Stretching Ratio	3.0／3.2	3.0／3.2	—

Table 3 Stretching Stress and Bubble Stability
<Main condition>

	Small scale research machine	Large scale product machine
Deformation rate	1.0	1.0
Stretching Stress (TD)	95 (MPa)	92 (MPa)
Bubble Stability	GOOD ±0.4 (%)	GOOD ±0.3 (%)

<Comparison condition>

	Small scale research machine	Large scale production machine
Deformation rate	0.6	0.5
Stretching Stress (TD)	75 (MPa)	68(MPa)
Bubble Stability	NOT GOOD ±0.8 (%)	NOT GOOD ±1.5 (%)

Table.4. Physical Property

		Small scale research machine	Large scale product machine
Deformation rate		1.0	1.0
Film impact strength		88,000 (J/m)	89,000 (J/m)
Penetrate strength		9.9 (N)	10.0 (N)
Tensile strength at break	MD	250 (MPa)	255 (MPa)
	TD	312 (MPa)	319 (MPa)
Tensile elongation at break	MD	126 (%)	129 (%)
	TD	113 (%)	110 (%)