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The Effect of Multiple Variables on Tensile Property of Injection-Molded Polypropylene Through the Combination of Orthogonal Design and Variance Analysis

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ABSTRACT: In this article, a combination of orthogonal design and variance analysis was used to study the systematical effect of vibration pressure, melt temperature, packing cycle, and mold temperature on the tensile property of polypropylene in dynamic packing injection molding (DPIM). The tensile measurement results show that all variables had significant influence on the final tensile property under the present molding condition. Meanwhile, the optimal molding condition to achieve high tensile strength was also obtained in the investigated ranges. Further quantitative statistical analysis revealed the degree of influence of four variables on the tensile property of polypropylene in DPIM. The sequence of the degree of influence from maximum to minimum is as follow: vibration pressure, melt temperature, packing cycle, and mold temperature. These findings provide the experimental and statistical evidences to illustrate that the application of orthogonal design and variance analysis might be an effective approach to systematically study the effect of multiple variables on the final mechanical property. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

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INTRODUCTION

Extensive studies have been performed on shear-induced crystallization under various kinds of flows in industrial and scientific fields, which led the molding techniques related to shear flow, such as dynamic packing injection molding (DPIM),[1–4] vibration extrusion,[5,6] and fiber spinning,[7] became popular in past several decades. It is well known that shear flow field causes the crystallization of polymers into oriented structures.[8–10] Generally, the so-called shish-kebab structure is formed under shear flow. This structure is very important from industrial viewpoint because it is structural origin of ultra-high strength and ultra-high modulus fiber. Although considerable researches have been performed in flexible polymers, only ultra-high modulus polyethylene fiber[11,12] has already been available in industry. One reason is that shear-induced molding process is very complicated, in which many factors have significant influence on the final microstructure that directly dominates the final mechanical property of molded products.

To date, some researches[13,14] regarding the effect of molding conditions on final property have been conducted in shear-induced molding techniques. The molding conditions included vibration pressure,[15] melt temperature,[16] mold temperature,[17] packing cycle,[18] etc. Chi and co-workers[19] have studied the effect of high-shear rate on mechanical property of iPP/PC blends. Their research results showed that the tensile strength of iPP composites significantly increased at high-shear rate. Bao and Tjong[20] prepared polypropylene nanocomposites via injection molding to study the effect of temperature and strain rate on the tensile behavior, and they found that tensile behavior highly depended on shear rate and temperature. Pantani et al.[21] studied the effect of packing pressure on morphology distribution in injection molding. Their results indicated that on increasing holding pressure, the molecular orientation inside the samples increased with the longer relaxation time caused by the higher pressures. Therefore, the final morphologies and properties were greatly influenced by packing pressure. However, so far, all researches almost focused in the effect of individual variable on the final mechanical property and not in the systematical effect caused by multiple factors.

Table I. Parameters of P, t, T_1 and T_2

Parameters	P (MPa)	T_1 (°C)	t (s)	T_2 (°C)
	9	170	5	20
	10	185	6	35
	11	200	8	50
	12	215	9	65
	13	230	11	80

So far, the systematical effect of multiple variables on the final mechanical property has seldom been considered because of its complexity, so it is not fully understood. Once the complex effect caused by multiple variables is known in injection molding, the morphology of polymers can be controlled by adjusting the experimental condition during processing, and then the products with excellent final property will be obtained for different flexible polymers. Under this situation, it is very necessary to perform systematical study regarding the effect of multiple variables on the final mechanical property.

In this work, DPIM technology was introduced to study the systematical effect of several main variables on the final tensile properties. Meanwhile, four molding variables, containing vibration pressure, melt temperature, packing cycle, and mold temperature, were chosen as research objects. For these variables, the estimation of the influence on tensile property will be drawn with the aid of orthogonal design and variance analysis based on the tensile measurements. In addition, the possible optimal molding condition will also be discussed based on the systematical analysis.

Generally, the application of orthogonal design may not only avoid many experiments, but also guarantee the scientificity of these experiments. However, to further quantitatively analyze the orthogonal experimental results, another statistical tool must be used. In this work, variance analysis as an effective tool in statistical analysis has been applied. Both orthogonal design and variance analysis have been detailed in Refs. 22–25.

EXPERIMENTAL

Material

The isotactic polypropylene (trademarked as F401, Lan Zhou Petroleum of China) was used in this experiment. It was a kind of commercial product with a melt flow index of 2.5 g/10 min.

Experimental Protocol

Four main variables, containing vibration pressure (P), packing cycle (t), melt temperature (T_1), and mold temperature (T_2), were presented in Table I. Twenty-five groups of experiments were designed by orthogonal design, as shown in Table II.

Sample Preparation

iPP melt was injected into a mold with the aid of an HT 100 g injection-molding machine with a barrel temperature of 180°C and an injection pressure of 6 MPa. Then DPIM technology was applied to introduce oscillatory shear to the cooling melt during packing stage by two pistons that moved repeatedly under the experimental condition (Table II). The detailed experiment procedure was described in Ref. 26. The layout of samples with main dimensions is shown in Figure 1. A molten pool is like an elastic spring to induce compression and expansion of melt in a runner of this experimental device.

Tensile Measurement

The tensile measurements were performed with the aid of testing machine (No. 4302) manufactured by America Instron (Norwood, MA). The moving speed of crosshead was 50 mm/min for tensile strength measurements. These measurements were performed at 23°C. Each tensile strength listed in Table II is an average value. To produce an average tensile strength and a standard deviation, seven tests were performed for each group of sample.

RESULTS AND DISCUSSION

Tensile Measurement Results

Table II shows the experimental conditions (the left side) and corresponding tensile measurement results (the right side). It should be noted that all the tensile strengths for DPIM specimens are higher than those of commonly molded samples. Meanwhile, one can observe that these values are very scattered with changing t, T_1, and T_2 at the constant P. For example, at P of 9 MPa, the maximum in tensile strength is about 61 MPa,

Table II. The Experimental Conditions and Corresponding Tensile Measurement Results

	Factors				
Order	P (MPa)	T_1 (°C)	t (s)	T_2 (°C)	Average tensile strength (MPa)
1	9	170	5	20	41
2	9	185	9	65	61
3	9	200	6	35	60
4	9	215	11	80	41
5	9	230	8	50	50
6	10	170	9	35	40
7	10	185	6	80	42
8	10	200	11	50	55
9	10	215	8	20	46
10	10	230	5	65	39
11	11	170	6	50	42
12	11	185	11	20	41
13	11	200	8	65	41
14	11	215	5	35	46
15	11	230	9	80	40
16	12	170	11	65	49
17	12	185	8	35	42
18	12	200	5	80	40
19	12	215	9	50	47
20	12	230	6	20	38
21	13	170	8	80	53
22	13	185	5	50	39
23	13	200	9	20	53
24	13	215	6	65	37
25	13	230	11	35	43

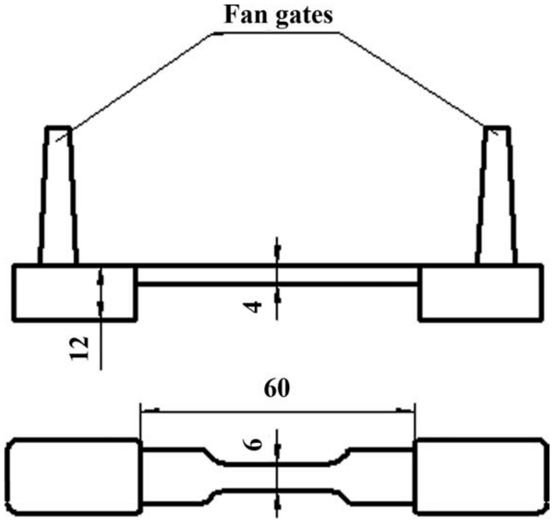

Figure 1. Schematic representation of injection-molded specimens (unit: millimeter).

but the minimum is only 41 MPa at the same *P*. The similar scatter phenomenon in tensile strength can also be found at the constant *t*, T_1, or T_2 whereas the parameters of other variables are changing. These results illustrate that the tensile strengths cover a wide range with the changing parameter at the constant variable *t*, T_1, T_2, or *P* in this experiment.

An Influence of Each Variable on the Tensile Property

To take the whole effect into account for each variable in DPIM, the fluctuation in a sum of tensile strength at each variable has been analyzed in this experiment. The sum of tensile strengths as functions of *P*, *t*, T_1, and T_2 were plotted in Figure 2. Figure 2(a) shows that the sum of tensile strengths goes down with increasing oscillation pressure at the initial stage, and then it is almost unity with the increase of oscillation pressure. Figure 2(b–d) shows the same tendency with the change of corresponding variable. At the first stage, the sum of tensile strengths significantly goes up with corresponding increasing melt temperature, packing cycle, and mold temperature, and then it rapidly falls down at the second stage even the parameter of the corresponding variable is still increasing. These results indicate that each variable had significant influence on the tensile property in the investigated ranges. Moreover, the change tendency of tensile property was very different between oscillation pressure and the other variables.

Optimal Molding Conditions in DPIM

After analyzing the change tendency of average tensile strength for each variable, the optimal molding condition can be obtained in the investigated ranges. The average tensile strengths with error bars for each variable were figured out and plotted in Figure 3. For vibration pressure, melt temperature, packing cycle, and mold temperature, the corresponding maximum in average tensile strength is 51, 50, 49, and 50 MPa, respectively.

Here, three characteristic regions can be seen according to the apparent difference of average tensile strength, as shown in Figure 3. In region I, the tensile properties $T_p \approx T_t \approx T_{T1} \approx T_{T2}$ (*T* and subscript represent tensile property and corresponding variable, respectively.) were about 50 MPa. These maximums of tensile property are very scattered in the investigated ranges. All

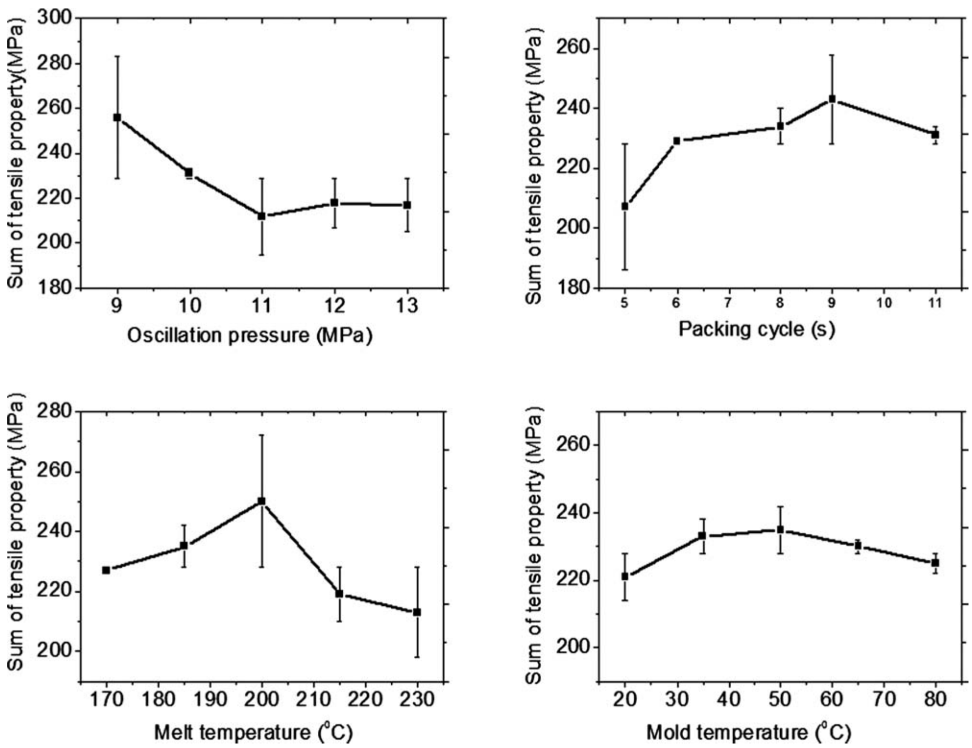

Figure 2. The sum of tensile strengths as functions of (a) oscillation pressure *P*, (b) packing cycle *t*, (c) melt temperature T_1, and (d) mold temperature T_2.

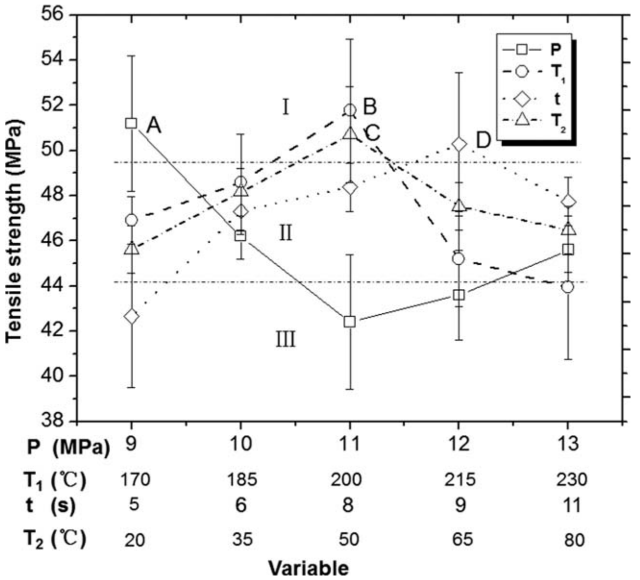

Figure 3. The average tensile strength as functions of (a) oscillation pressure P, (b) packing cycle t, (c) melt temperature T_1, and (d) mold temperature T_2.

of them are marked by A, B, C, and D, as shown in Figure 3. A, B, C, and D represents that P, T_1, T_2, and t were 9 MPa, and 200°C, 50°C and 9 s, respectively. This condition might be the most possible optimal molding condition in present experiment. It should be noted that, here, the optimal molding condition is only limited in our current investigated ranges. In regions II and III, the average tensile strengths are significantly less than those in region I. This means that, in these regions, products with excellent tensile property cannot be obtained. Especially in region III, the average tensile property is about 42 MPa, which is nearly equal to that of commonly molded sample.

According to the results mentioned above, the experimental results obtained based on orthogonal design can systematically reflect the change tendency of tensile property even when there are multiple variables. This suggests that the application of orthogonal design should be reasonable to study the systematical effect caused by multiple variables on the final mechanical property in injection molding.

Variance Analysis

To quantitatively analyze the effect of several variables on the final tensile property, the so-called variance analysis was introduced to measure the degree of influence of four variables in present experiment. Variance analysis is widely used to analyze the degree of influence of each variable when the changing variables are multiple in science research, which highly depends on the preceding experiment performed under orthogonal design. Variance is calculated by the following equation:

$$S^2 = (1/n)[(x_1 - m)^2 + (x_2 - m)^2 + ... + (x_n - m)^2]$$

Here, we define S_w is random error, and S_b is experimental error. The total error

$$S_t = S_w + S_b.$$

A calculation is performed through the univariate process that goes on automatically based on a computer program. Before calculating, experimental results have been obtained based on the orthogonal design. In variance analysis, an observed power α is the most important parameter to judge the degree of influence for every variable on the final tensile property. Meanwhile, α can be used to judge whether the variable had some effect on the tensile result or not. If α is greater than the given α value, this suggests that the fluctuation of property is reasonable in the investigated range. Otherwise, it is unreasonable. In this work, two standard α values (0.005 and 0.01) were chosen as the compared criterion. The analysis results are listed in Table III. When the criterion α is 0.005, the corresponding observed powers of P, t, T_1, and T_2 are 0.025, 0.019, 0.016, and 0.007, respectively. At the criterion α of 0.01, the corresponding observed powers of P, t, T_1, and T_2 are 0.046, 0.035, 0.030, and 0.013, respectively. It is very obvious that all the observed α are far beyond the corresponding given alpha value, suggesting that each variable has significant effect on the tensile property in this experiment, which accords with the results mentioned in section 3.2. Based on these α values, the influence sequence of several variables on the tensile property is as follow: vibration pressure > melt temperature > packing cycle > mold temperature.

CONCLUSIONS

The effect of four main variables, containing oscillation pressure, melt temperature, packing cycle, and mold temperature, on the final tensile property has been studied through the combination of orthogonal design and variance analysis in DPIM. All the experiment results are presented above.

Table III. Variance Analysis Result at Observed Powers of 0.005 and 0.01 (Dependent Data: Tensile Strengths)

Source	Type III sum of squares	df	Mean square	F	Sig	Partia Eta square	Noncent parameter	Observed Power (α) 0.05	Observed Power (α) 0.1
P	222.317	4	55.579	0.838	0.538	0.295	3.352	0.025	0.046
T_1	169.451	4	42.363	0.639	0.65	0.242	2.555	0.019	0.035
t	141.636	4	35.409	0.534	0.715	0.211	2.135	0.016	0.030
T_2	24.734	4	6.183	0.093	0.982	0.045	0.373	0.007	0.013

1. All the chosen variables had significant influence on the final tensile property under the experimental condition. Based on the systematic analysis, the optimal molding condition to obtain products with excellent tensile property in DPIM has been obtained in present investigated ranges.
2. The quantitative variance analysis shows that the influence sequence of several variables on the final tensile property in DPIM is as follow: vibration pressure > melt temperature > packing cycle > mold temperature.
3. It might be an effective approach to analyze the complex influence of multiple variables on the final mechanical property through the combination of orthogonal design and variance analysis.

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