

Taguchi analysis of shrinkage and warpage of injection-moulded polypropylene/multiwall carbon nanotubes nanocomposites

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Received 25 May 2009; accepted in revised form 26 July 2009

Abstract. This paper focuses on the effect of multi-walled carbon nanotube (MWNT) addition on shrinkage and warpage properties of polypropylene (PP) injection mouldings before and after annealing. A Taguchi design of experiments has been implemented to highlight the influence and optimise processing conditions such as injection flow rate, holding pressure, back pressure and screw speed. The addition of 2 wt% of carbon nanotubes into PP significantly reduces the shrinkage and warpage of injection-moulded parts as compared to the neat PP. Shrinkage reduction up to 48% (respectively 33%) is noticed in the flow direction before (respectively after) annealing, whereas warpage reduction exceeds 55%. The sensitivity of the injection-mouldings dimensional properties to processing parameters remains roughly the same in case of neat PP and PP/MWNT nanocomposites when shrinkage is considered. It is even significantly reduced by carbon nanotubes addition when warpage is considered. Furthermore, the Taguchi method provides an efficient and effective tool to study the effects of process parameters on the warpage and shrinkage of injection moulded parts. The additive model used works well for predicting the warpage and shrinkage behaviour of PP and PP/MWNT composites.

Keywords: nanocomposites, polypropylene, multi-walled carbon nanotubes, Taguchi analysis, shrinkage

1. Introduction

Carbon nanotubes (CNT) are promising fillers for polymer materials due to their outstanding mechanical, electrical, and thermal properties [1–4]. These new nanocomposite materials find industrial application in the field of electrostatic dissipation [5], electromagnetic interference-shielding (associated with both commodity polymers [6] and engineering polymers [7]), and electrically conductive materials achieving at the same time enhanced stiffness, strength, impact properties, thermal stability, tribological properties, and reduced thermal expansion

[8]. Further, the manufacturing of industrial parts from CNT/polymer nanocomposites requires the development of processing technologies, which are preferably compatible to already existing industrial moulding technologies. Moreover, a good knowledge of the relationship between processing conditions and properties of the nanocomposites is crucial. Most of the reported literature on nanotubes filled polymers has focussed on the quantitative [9] or qualitative [10] description of the dispersion state of nanotubes, the improvement of nanotube dispersion by polymer [11] or nanotubes

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[12] modifications and/or proper selection of processing parameters (e.g. extrusion rotation speed and screw design [13], shear stress [14]), also on structure [15], viscoelastic (both in solid [16] and molten [17] state), electrical (conductivity [18], resistivity [19], percolation threshold [20]), mechanical (at room [21] and high [22] temperature) or interfacial [23] properties, as well as gas permeability and thermal stability [24] of the CNT filled polymers nanocomposites.

However, industrial parts specifications and requirements are not limited to the above mentioned usage properties. The dimensional accuracy and stability of the parts are often important quality and functional criteria (especially when parts are assembled to build more complex structures). Nevertheless, systematic investigations on the influence of nanofiller addition and of process conditions on dimensional properties of polymer nanocomposites and their variation as a function of time have not been addressed in the literature up to now. Salahuddin and Shehata [25] have studied the very particular case of polymerisation shrinkage and warpage of heat-cured dental acrylic resin. These authors have shown that addition of nanoclay reduces dimensional and shape variations, but without having investigated the effect of processing parameters modifications.

Injection moulding is one of the most important polymer processing methods for manufacturing of plastic parts. The plastic part obtained by injection moulding process is approximately the shape of the mould and may not involve further operation. The final dimensions and geometry of the moulded part is usually a primary concern in terms of manufacturing quality. Because of some physical aspect of the process, significant deviations are observed between the dimensions and shape of the cavity and those of the final parts. Those discrepancies are caused by thermal effects such as difference between the linear thermal expansion coefficients of the mould and the polymer [26], crystallization and thermal kinetics [27], flow effects inducing filler [28] and molecular orientation gradients [29, 30], as well as mould deformations [31]. Practically, the deviations from the mould geometry may be described by shrinkage and warpage, shrinkage regarding dimensional differences and warpage regarding shape differences [32]. To evaluate and improve achievable tolerances of injection moulded

parts, the effects of the outlined phenomena have to be quantitatively assessed. Therefore, it is of critical importance to effectively control the factors (e.g. temperature [30], mould geometry [33], holding pressure and injection velocity [34], holding pressure and packing time [35]), which influence the moulding process from this standpoint.

Also, it is known that the annealing of thermoplastics after the manufacturing causes changes in the structure and properties of the materials. This process has been employed to improve the final properties of the polymers via healing of defects and diminishing residual stress and strain. In addition, lamellae thickening and rearrangement of the chain during heating may also occur upon annealing [36]. Therefore, it would be worth considering also the effect of annealing on the dimensional properties of the nanocomposites parts.

Since a large number of parameters influences injection-moulding processes, a full-factorial experiment is required to determine the important factors and optimal process conditions. This is both costly and time consuming. Therefore, design of experiments (DOE) is usually adopted to schedule the injection experiment [37, 38]. DOE commonly uses the method invented by Taguchi [39].

In that context, this paper proposes an expansion of our previous research on nanotube filled polypropylene nanocomposites [11, 16]. It aims at systematically investigating the effects of nanofiller addition and variation in process conditions such as holding pressure, back pressure, injection flow rate, and screw rotation speed, on the shrinkage (along- and across-the-flow directions) and warpage of injection-moulded PP/MWNT nanocomposite in U box shaped parts on the basis of a Taguchi DOE. This work therefore constitutes a first step of the future development of guidelines for plastics converters.

2. Materials and methods

2.1. Materials

Polypropylene (PP) – multi wall carbon nanotubes (MWNT) nanocomposites were produced by mixing in a co-rotating twin screw extruder (Clextral, Firminy, France) at a barrel temperature of 195–210°C and a screw speed of 50 rpm homo PP granules (Polychim polypropylene with a melt flow index of 12 g/10 min at 190°C, Polychim Industrie, Loon-Plage, France) with a commercial master-

batch containing 20 wt% of MWNT compounded by extrusion process (Plasticyl-2001 supplied by Nanocyl, Sambreville, Belgium). The specifications of MWNTs in the masterbatch are as follows: average diameter is 10 nm, average length of the nanotubes is 1.5 μm , and purity > 90%. During melt extrusion ventilation was kept on to remove trapped air in the compounds. After pelletizing, the nanocomposite granules were dried in a vacuum oven at 120°C for at least 4 h before injection-moulding. Previous studies from our laboratory indicated that incorporation of 2 wt% of MWNTs in PP gives better mechanical properties [16]. Therefore, 2 wt% of MWNTs filled PP nanocomposites have been chosen for the present study.

2.2. Injection moulding conditions

The injection moulding machine used had a clamping force of 800 kN (Krauss Maffei, Munich, Germany) and was equipped with a specially designed 'U' box type mould. The injection-moulded plastic part manufactured had a 2 mm uniform thickness and its 'U' shape roughly reproduces the geometry of many industrial parts (boxes, bumper, dash board insert, etc.). The mould cavity was fed by a sprue gate as show in Figure 1. The cavity was equipped with a melt pressure sensor that was used to control the process repeatability.

The investigations carried out mainly aimed at determining the influence of the chosen input parameters (injection moulding conditions) on the output quantities (such as shrinkage and warpage), and further using experimental techniques and statistical methods for the data analysis, to establish the relationship between them in the form of a function. Taguchi design of experiments (DOE) have been used for this purpose. The experiments consist

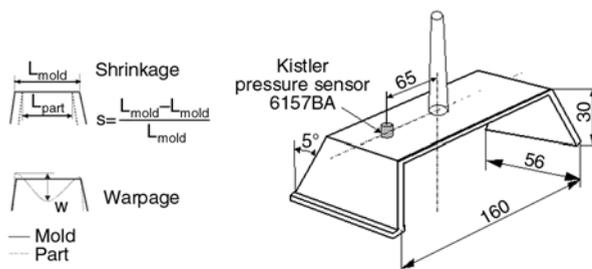


Figure 1. Typical part manufactured by injection moulding

Table 1. Factors and levels selected in the DOE

Factors	Level 1 (low)	Level 2 (high)
Injection speed [cm^3/s]: Q	25	50
Holding pressure [bar]: hP	250	350
Back pressure [bar]: bP	35	65
Screw rotational speed [rpm]: Ω	50	90

Table 2. L16 (24)orthogonal array used for the DOE

Trial No	Q	hP	bP	Ω
1	1	1	1	1
2	1	1	2	2
3	1	2	1	2
4	1	2	2	1
5	2	1	1	2
6	2	1	2	1
7	2	2	1	1
8	2	2	2	2
9	1	1	1	2
10	1	1	2	1
11	1	2	1	1
12	1	2	2	2
13	2	1	1	1
14	2	1	2	2
15	2	2	1	2
16	2	2	2	1

of two identical L16 orthogonal array fractional factorials designs, the first one with the neat PP and the other with the PP/MWNT nanocomposite. Four processing parameters, which were expected to be significant in their effects on the induced microstructure and the properties of the injection-moulded parts were selected as variables in these DOE: injection speed or volume flow rate (Q), holding pressure (hP), back pressure (bP) and screw rotational speed. Because thermal parameters have a very high effect on part stability [40], taking into account melt and mould temperatures in the experimental design would not have permitted to investigate with a sufficient accuracy the influences of the four other processing parameters. Each studied parameter was set up at two levels (low and high) according to the experimental matrix shown in Table 1. The other processing parameters were kept constant: mould temperature was kept at 35°C and polymer melt temperature at 210°C; cooling time was maintained at 25 seconds, and a holding time of 35 sec was fixed. The L16 fractional orthogonal design array and the levels of set-up conditions are reported in Table 2.

2.3. Shrinkage and warpage measurements

During the injection moulding experiments, each process condition was allowed to stabilize for at least half an hour. After that, ten parts were moulded at each process condition. The sixth to tenth injection-moulded parts were further characterized. Shrinkage and warpage were measured one week after moulding and conditioning at 23°C and 50% HR so as to stabilise the material.

Annealing of the injection-moulded parts was carried out at a temperature of 120°C during 5 hours, followed by a cooling at 23°C and 50% relative humidity (henceforth this condition will be referred as annealed one) so as to allow residual stresses relaxation without inducing structural change in polypropylene [36].

The shrinkage and warpage of the injection-moulded parts are defined in Figure 1. Mould shrinkage S was measured as per the ISO-294-4 standard. S is the relative difference between the dimensions of the part and the dimensions of the mould. It is expressed as a percent change in dimension of a specimen in relation to mould dimensions. The same procedure is used for annealed samples. Generally, shrinkage of many materials differs for flow (S_F) and transverse (S_T) (or across flow) directions. Flow direction is taken as the direction the molten material is travelling when it exits the gate and enters the mould.

Mould shrinkage in the flow direction is calculated by Equation (1):

$$S_F = 100 \cdot \frac{L_M - L_P}{L_M} \quad (1)$$

where L_M is the length of the mould cavity and L_P is the corresponding length of the part after it has cooled.

Mould shrinkage in the transverse direction is calculated by Equation (2):

$$S_T = 100 \cdot \frac{W_M - W_P}{W_M} \quad (2)$$

where W_M is the width of the mould cavity and W_P is the corresponding width of the part after it has cooled.

Warpage measurements were carried out by using a Cyclone 3D measurement machine. The values of warpage are measured as the displacement difference between upper and lower points on the injection

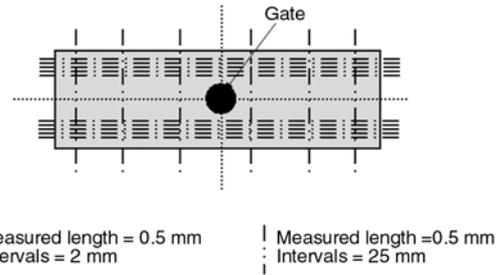


Figure 2. Warpage measurement spots of injection-moulded parts

tion moulded part surface at ten different longitudinal lines as shown in Figure 2.

3. Results and discussion

3.1. Shrinkage

The mean values of shrinkage in both flow and transverse directions of all samples from the 16 runs for PP and PP/MWNT nanocomposites were measured before annealing (Figures 3a and 4a) and after annealing (Figures 3b and 4b). Whatever the material may be (neat PP or PP/MWNT nanocomposite), it undergoes logically on the average between 2 and 4 times more shrinkage in transverse direction than along the flow direction. This may be explained by the combined effect of crystallization and flow-induced molecular and/or filler orientations, which is in accordance with the literature [34].

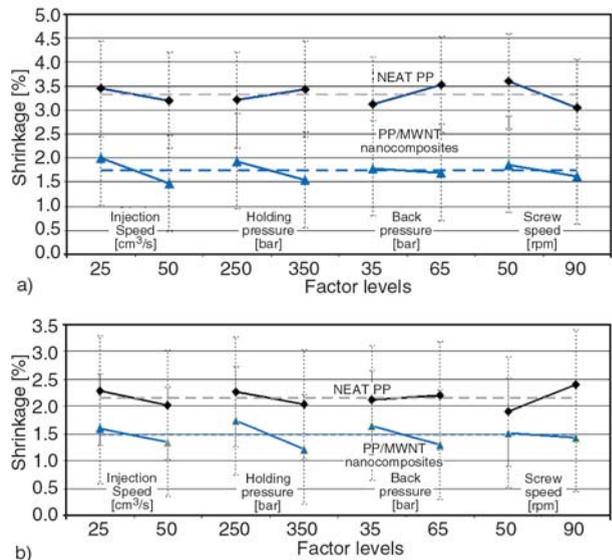


Figure 3. Shrinkage of PP and PP/MWNT nanocomposites in flow direction before annealing (a) and after annealing (b) – Standard deviations represent data scattering when the considered factor is set up to its low or high level in the Taguchi DOE

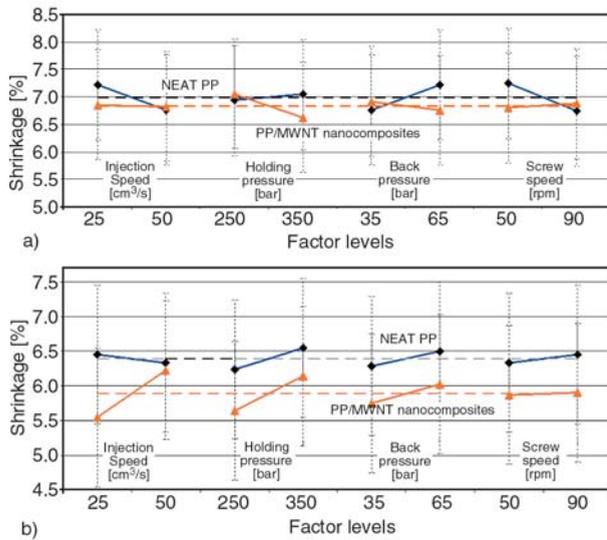


Figure 4. Shrinkage of PP and PP/MWNT nanocomposites in transverse direction before annealing (a) and after annealing (b) – Standard deviations represent data scattering when the considered factor is set up to its low or high level in the Taguchi DOE

The addition of 2 wt% of MWNTs into PP matrix allows reducing significantly the moulding shrinkage of injection moulded-parts. Compared to neat PP, this reduction reaches 48% on the average before annealing and 33% after annealing in the flow direction. The carbon nature of the nanofiller used highly limits its sensitivity to thermal expansion in the temperature range considered during injection moulding. Carbon nanotubes are not subjected to shrinkage and therefore limit the nanocomposite shrinkage accordingly. The shrinkage reduction effect induced by addition of carbon nanotubes is slightly less after annealing. This may be mainly ascribed to the completion of the relaxation mechanisms undergone by the neat PP after annealing.

The effect of 2 wt% of MWNTs addition into PP is much lower in the transverse direction even if still significant, as the shrinkage reduction is limited to about 5% before annealing and about 10% after annealing.

The influence of the processing parameters (injection speed, holding pressure, back pressure and screw rotation speed) are much more difficult to handle. Actually, the investigated processing parameters sometimes induce a quite low variation in the measured dimensional property. Therefore it makes no sense to discuss the dependence of the shrinkage on each moulding parameter in that case. It is how-

ever worth noting that the sensitivity of the injection-mouldings shrinkage to significant processing parameters globally remains roughly the same (variation of output parameters with similar orders of magnitude) for neat PP and PP/MWNT nanocomposites (except in one case, e.g. effect of injection speed on the transverse shrinkage after annealing; the reason for this exception has not been elucidated).

3.2. Warpage

Apart from shrinkage, warpage behaviour of injection-moulded parts is crucial in determining the polymer dimensional accuracy and subsequent application. Warpage behaviour of PP and PP/MWNT materials is shown in Figure 5. Nanotubes addition in PP clearly reduces average warpage by more than 55% both before and after annealing.

Warpage is known to be related to the existence of residual stresses related to heterogeneous temperature and cooling rate distributions, non-symmetric processing-induced morphology and orientation patterns through the part thickness. Under the same set of processing conditions in a Taguchi DOE, Fourdin *et al.* [41] have shown that nanofillers tend to make the injection-moulded parts morphology much more homogeneous, the nucleation being favoured instead of crystal growth during the crystallisation process. This reduced material heterogeneity then limits the local differential shrinkage

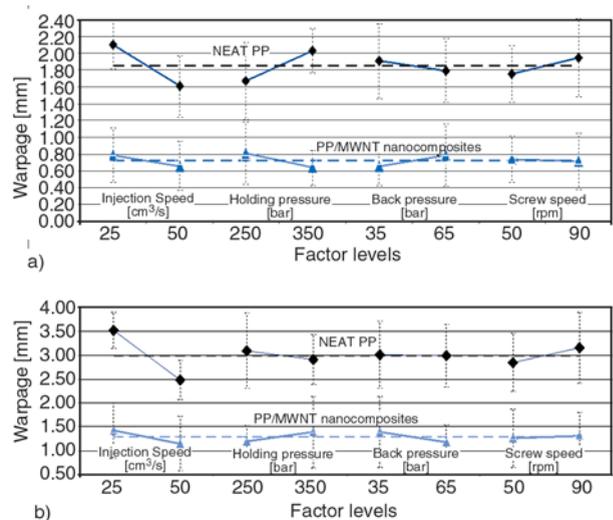


Figure 5. Warpage of PP and PP/MWNT nanocomposites before annealing (a) and after annealing (b) – Standard deviations represent data scattering when the considered factor is set up to its low or high level in the Taguchi DOE

and therefore the parts warpage. Another reason for the warpage reduction noticed upon carbon nanotubes addition may also be that the higher the rigidity of the material is, the less the part is able to deform (addition of 2 wt% of carbon nanotubes into PP increases its modulus by 35% as reported in our previous paper [16]).

Moreover, the warpage in PP/MWNT sample is less sensitive to the variation of the moulding parameters when compared to PP parts, where injection speed (primarily) and holding pressure (in a lesser extent) appear to affect significantly the part warpage. This suggest that the geometrical shape of nanocomposite injection-moulded part is less sensitive to fluctuation of processing parameters than neat PP ones. The use of nanocomposites therefore improves the robustness of the injection moulding process.

3.3. Verification tests

Additive model is being used in Taguchi method in order to predict the influence of the control factors on the response [39]. The model refers to the sum of the individual factor effects with cross-terms (interactions). One major purpose of the verification experiment is to provide evidence that shows the additive equation applies and that interactions are low. The applicability of the additive model cannot be shown simply from the factor effects plots. The general form of the predictive equation is given by Equation (3):

$$Y_{pred} = Y_{expt} + a(Q) + b(hP) + c(bP) + d(\Omega) + e(Q - hP) + f(bP - \Omega) \tag{3}$$

where Y_{pred} is the predicted value, and Y_{expt} the overall average response for the orthogonal array. Factors a, b, c and d are coefficients (low level for optimal combination or high level for minimal

combination) of corresponding parameters. ‘ e ’ and ‘ f ’ are the coefficients of interaction parameters for ‘ $Q-hP$ ’ and ‘ $bP-\Omega$ ’ respectively. All these values are obtained by analysis of variance and are tabulated in Table 3.

The factor effects corresponding to the factor levels being modelled (typically the optimum levels) are used in the predictive equation. Since the quality of warpage and shrinkage is the type of ‘Smaller the Better’, the optimal combination of studied factors for the smallest warpage and shrinkage can be identified from the response plots.

Table 4 summarizes the optimal factor combinations for the PP and PP/MWNT materials. The predicted optimal warpage and shrinkage (along flow and across flow direction) of each case is obtained by substituting the corresponding factors’ effects shown in the response plots into the predictive equation. In practice it is very difficult to state with precision how close the experimental numbers must come to the predicted values for the agreement to be considered good. This model holds good only for this present set of parameters, but it validates the previous statistical analysis. As shown in Table 4, all the predicted values fall within the range of standard deviation of experimental means, except for the predicted value for PP/MWNT warpage before annealing.

4. Conclusions

The addition of 2 wt% of carbon nanotubes into PP significantly reduces the shrinkage and warpage of injection-moulded parts as compared to the neat PP, both before and after annealing. Shrinkage reduction up to 48% (resp. 33%) were noticed in the flow direction before (resp. after) annealing, whereas warpage reduction was found to exceed 55%. The extent of anisotropic shrinkage across-

Table 3. Model coefficient values calculated from variance analysis

Dimensional properties		Warpage				Shrinkage							
		Before annealing		After annealing		Flow direction				Transverse direction			
		PP	PP-MWNT	PP	PP-MWNT	PP	PP-MWNT	PP	PP-MWNT	PP	PP-MWNT	PP	PP-MWNT
Model coefficients	a	-0.2465	-0.0645	-0.5250	-0.1362	-0.1240	-0.2644	-0.2306	-0.1198	0.2307	-0.0146	-0.0609	-0.3403
	b	-0.2781	-0.0851	-0.0900	-0.0969	-0.1170	-0.1903	-0.2159	-0.2622	-0.0535	-0.2141	-0.1546	-0.2487
	c	-0.0561	-0.0635	0.0069	-0.1046	-0.2049	-0.0418	0.0413	-0.1747	-0.2269	-0.0778	-0.1103	-0.1340
	d	-0.0966	-0.0138	-0.1583	-0.0282	-0.2749	-0.1229	-0.2612	-0.0358	-0.2517	-0.0363	-0.0603	-0.0166
	e	-0.0813	-0.0022	-0.1585	-0.0783	-0.1748	0.0042	-0.1999	-0.0412	-0.1981	-0.0971	-0.1193	-0.3660
	f	-0.0074	-0.1168	-0.0411	-0.1102	-0.3068	-0.0890	-0.1233	-0.1425	-0.1891	-0.0499	-0.2448	-0.1388

Table 4. Verification conditions, predicted values and experimental values

Dimensional properties		Warpage				Shrinkage							
		Before annealing		After annealing		Flow direction				Transverse direction			
		PP	PP-MWNT	PP	PP-MWNT	Before annealing		After annealing		Before annealing		After annealing	
PP	PP-MWNT					PP	PP-MWNT	PP	PP-MWNT	PP	PP-MWNT		
Processing parameters (Optimal combination)	Injection speed [cm ³ /s]	50	50	50	50	25	50	50	50	50	50	50	25
	Holding pressure [bar]	250	350	250	250	350	350	350	350	350	350	250	250
	Back pressure [bar]	35	35	35	65	35	35	35	65	35	35	65	65
	Screw rotational speed [rpm]	50	90	50	50	90	50	90	50	50	50	50	90
Predicted values for warpage [mm] and shrinkage [%]		1.807	0.379	2.238	0.733	2.126	1.104	1.164	0.697	6.303	6.346	5.643	4.643
Experimental values for warpage [mm] and shrinkage [%]		1.064 ±0.018	0.342 ±0.027	2.108 ±0.082	0.702 ±0.007	2.285 ±0.028	1.002 ±0.071	1.165 ±0.052	0.665 ±0.034	6.152 ±0.020	6.375 ±0.146	5.831 ±0.035	4.142 ±0.011

the-flow direction is logically more than the shrinkage along-the-flow direction. The sensitivity of the injection-mouldings dimensional properties to investigated processing parameters remains roughly the same in case of neat PP and PP/MWNT nanocomposites when shrinkage is considered. It is even significantly reduced by carbon nanotubes addition when warpage is considered.

Furthermore, the Taguchi method provides an efficient and effective tool to study the effects of process parameters on the warpage and shrinkage of injection moulded parts. The additive model works well for predicting the warpage and shrinkage behaviour of PP and PP/MWNT composites.

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