MATERIALE PLASTICE
https://revmaterialeplastice.ro
https://doi.org/10.37358/Mat.Plast.1964

Shrinkage and Warpage in the Permanent Shape of Sape-Memory Polyurethane Parts

SUKRAN KATMER[1], CETIN KARATAS[2*]
[1] Graduate School of Natural and Applied Sciences, Gazi University, 06500 Teknikokullar, Ankara, Turkey
[2] Manufacturing Engineering, Faculty of Technology, Gazi University Teknikokullar, Ankara, Turkey

Abstract: *The shape memory effect, as the most important ability of shape memory polymers, is a working property and provides the design ability to shape memory polymer features. Shrinkage and warpage are important parameters to control the dimensional accuracy of permanent and temporary shapes of an injection moulded shape memory polyurethane (SMPU) part. In this study, the effects of injection moulding parameters on the shrinkage and warpage of the permanent shape of moulded SMPU parts were experimentally investigated. The parameters of injection pressure, melt temperature, mould temperature, packing pressure, packing time, and cooling time, were chosen as the injection moulding control factors. Taguchi's L27 orthogonal array design table was used with six injection moulding parameters and their three levels. The results showed that the part has different shrinkage ratios in three main directions, namely, the flow direction, perpendicular to the flow direction, and the direction through the thickness. The results of the analysis of variance showed that the cooling time is the most influential parameter on both the shrinkage (except in thickness) and warpage. The shrinkage in the flow direction as well as in perpendicular to the flow direction decreased with increasing the cooling time. Warpage also decreased with increasing the cooling time. Injection pressure and melt temperature were found to be effective on shrinkage in thickness. Effects of mould temperature, packing pressure, and packing time were found to be limited. A statistically significant relationship has been noticed among shrinkage, warpage, and residual stresses during the study.*

Keywords*: shape memory polyurethanes, injection moulding, permanent shape, shrinkage, warpage*

1. Introduction

Shape memory polymers (SMPs) provide two or more working shapes in one part as different from general-purpose polymer materials. Their shape memory effect (SME) makes them usable in various shapes in a part and doing a work while shape-changing when facing stimuli such as temperature, *p*H, light, electric current, magnetic field, etc. [1-3]. Recently, shape memory polymers and their composites have been used to manufacture parts and they are featured in large application areas such as medical applications [4-8, 1, 9], self-disassembly systems [10-13], or aviation and space applications [14-16]. Due to their shape-changing property, the precise controlling of the permanent and temporary shapes of manufactured or moulded shape memory parts is critical for a system. In these applications, shape memory polyurethanes (SMPUs) have also many applications; for instance; fasteners [13], bolts [10], orthodontic wires [8], braided stents [17], artificial muscles [9], etc.

It is well known that moulding methods have a significant influence on moulded parts. As the most common plastic processing method, injection moulding parameters have influences on residual stresses [18-20], shrinkages [21-23], warpages [24-28], dimensional accuracy [22], etc. of moulded plastic parts. Many studies were carried out on the effects of injection moulding process parameters on various plastic and plastic composite materials. In an experimental study, cooling time was determined as the most influential moulding parameter on residual stresses in moulded shape memory polyurethane (SMPU) parts and followed by packing pressure, mould temperature, and injection pressure [19]. The reason for residual stresses during the injection stage was described as high shearing stresses that occur during the filling stage in melted plastic at lower temperatures due to the rheological properties of the melt.

**email: cetink@gazi.edu.tr*

If the stresses do not have enough time for relaxation, then they can cause warpage in the part after demoulding [24]. In another study, it was reported that the shrinkage decreases on acrylonitrile butadiene styrene (ABS), polystyrene (PS), polycarbonate (PC), and high impact PS (HIPS) parts at high - level melt temperatures and packing pressures [23]. It was found that the shrinkage has a lower value when higher mould temperature and injection pressure is applied on ABS parts [21]. Another study revealed that the most influential moulding parameter on the dimensional accuracy of high-density polyethylene (HDPE) is packing pressure; and that the dimensional accuracy increases while the packing pressure is increased [22].

Taguchi's experimental design takes an important place in experimental studies in engineering applications because of the opportunity of reducing the number of experiments. The optimisation of injection moulding process parameters [29, 30] or investigation of effects of injection moulding process parameters on residual stresses [19, 31, 32], warpage [27, 28, 33-35], shrinkage [35, 36], short shot [19], or other part properties of different plastic materials, were investigated using Taguchi's designs [29, 37].

The studies discussed above show that injection-moulding conditions have different influences on the properties of moulded plastic parts depending on the types and properties of plastic raw materials. As an applicable polymer material in industrial applications, it should be understood, as to how the dimensional accuracy (dimensional properties) of SMPU parts can be changed by the moulding processes. For that reason, the injection moulding process of thermoplastic shape-memory polyurethane, was investigated experimentally, and this study focuses on the effects of moulding parameters on the shrinkage and warpage of injection moulded SMPU parts to depict and clarify as to how the permanent shape dimensional accuracy changes by the moulding conditions. According to the previous studies, the most powerful injection moulding parameters, which are injection pressure, melt temperature, mould temperature, packing pressure, packing time, and cooling time, were examined by using Taguchi's L27 orthogonal array design table. At the end of the study, the results showed that the moulding parameters have various influences on the shrinkage and warpage of SMPU parts.

2. Materials and methods

2.1. Material and moulding

A shape memory polyurethane (SMPU) material (Diaplex MM4520 type) was purchased from SMP Tech. Co. (Japan) and moulded by an injection moulding process using ARBURG All-rounder 220-300 (Germany) injection moulding machine as a tensile test bar-shaped part. The shape of the specimen and sprue, runner, and the cooling system of the mould are shown in Figure 1. The cooling channel was also in the same pattern on the other half of the mould. The material properties, which were provided by the material's manufacturer, are shown in Table 1.

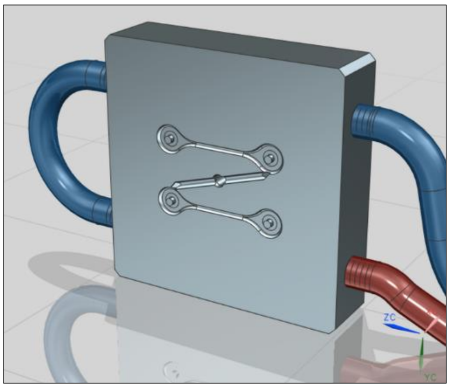

Figure 1. The stationary half of the injection mould

Table 1. Properties of Diaplex MM4520

Properties	Unit	Value	
		Glass region	Rubber region
Hardness	H_DD	76	30
Tensile Strength	MPa	55	10
Elongation	%	30-50	>600
Bending Modulus	MPa	2150	-
Bending Strength	MPa	80	-
Specific Gravity	g/cm^3	1.25	
Poisson Ratio	-	0.5	
Glass Transition Temperature (T_g)	K	318	
MFI (200°C, 5.00 kg)	g/10min	48	

2.2. The Design of experiments

For monitoring the effects of the whole injection moulding process from start to end, six moulding parameters and their three levels were chosen (Table 2). In Table 2 and Table 3, all parameters were indicated by capital letters from A to F, respectively. The information and suggestions on the material data sheet were considered in order to decide the boundary conditions for moulding. The packing pressure was applied as a function of the injection pressure (40, 50, and 60% of injection pressure). The packing and the cooling times were calculated by using the equations that were given in the literature [38].

Taguchi's experimental design is a method, which is based on statistical principles and uses orthogonal arrays for reducing the number of experiments. Taguchi method analyses the data by the variance of analysis (ANOVA) method and provides results as an ANOVA table, and the main-effect plots by signal to noise (S/N) ratio and the mean of data. S/N ratio is an indicator that is calculated by four different formulae according to the data. "*Larger is better*" formula is used when the response is wanted to be maximised. "*Nominal is best*" formula is used when the response is wanted to be a target. Finally, "*Smaller is better*" formula is used when the response is wanted to be minimised [39].

As the table of experiments, for six factors and their three levels, Taguchi's L27 (6^3) orthogonal array was applied (Table 3). All the results obtained from experiments were evaluated by ANOVA via Taguchi's design of experiment (DoE) analysis. In this study, S/N ratios were calculated via the formula named "*smaller is better*" for the shrinkage and the warpage.

Table 2. The design of the experiment

Factors	Levels		
	- (1)	0 (2)	+ (3)
(A) Injection pressure (MPa)	50	60	70
(B) Melt temperature (K)	468	473	478
(C) Mould temperature (K)	303	308	313
(D) Packing pressure (MPa)	A*40%	A*50%	A*60%
(E) Packing time (s)	6	9	12
(F) Cooling time (s)	15	22.5	30

2.3. Measurements of shrinkage and warpage

Figure 2 shows the part shape, and dimensions and the feeding system of the cavity. The dimensions which were chosen to control are L, the distance between holes (80 mm); G, the gauge diameter (Ø4 mm); d, the hole diameter (Ø6 mm), which is close to the gate; D, the outer diameter (Ø23 mm), which is close to the gate; and t, which is the thickness (4 mm). The warpage of the specimens was measured as explained in Figure 3. The shrinkage ratios were calculated for each dimension and analysed individually. The shrinkage was calculated as a percentage of the difference between the actual dimensions of moulded parts and the mould dimensions. The length and the width were defined as along and perpendicular to flow direction, respectively.

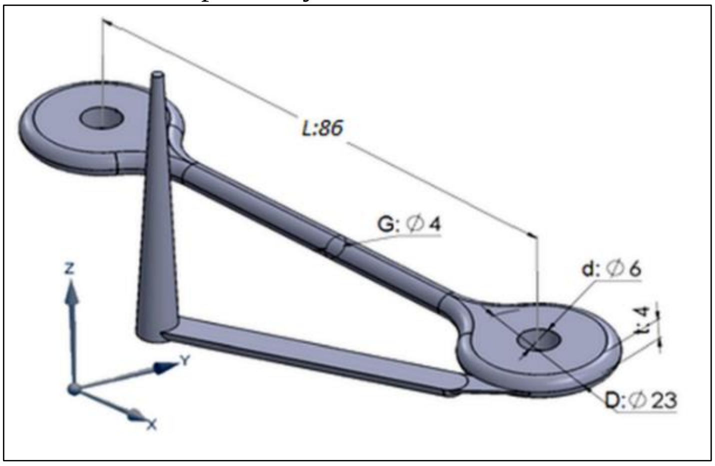

Figure 2. The runner system, the specimen shape and dimensions

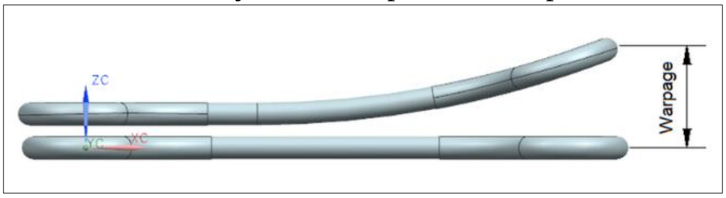

Figure 3. The representation of warpage

3. Results and discussions

Analysis of variance (ANOVA) and the main-effect plots, were used to evaluate the results by utilizing the mean values of the measured results. This paper includes only the mean results, since the results of the SN ratios and mean values show similar outputs. In the main-effect plots, if the line is more horizontal than vertical, it means that the parameter has a weak effect or no effect on the results. The study was built upon the hypothesis, which is named as $H_{0,}$ stating that there is no meaningful difference between the effects of the moulding parameters on shrinkages and warpage. The alternative hypothesis, H_1, is that at least one parameter has a different effect from the other parameters.

H_0: μA=μB=μC=μD=μE=μF

H_1: at least one average value (μj) is different from others.

F-test evaluation has been used to consider the results of which parameter has an important effect, statistically. $F_{critical}$: 3.37 was read from standard F tables using the total degree of freedom (26), factor`s degree of freedom (2), and α=0.05 and was compared to F-values of all parameters in all ANOVA tables. The experimental data of shrinkage and warpage on average were given in Taguchi's L27 design table in Table 3.

3.1. ANOVA results

The results table of the ANOVA of shrinkage ratios and warpage are shown in Table 4 and Table 5. The F distribution is a right-skewed distribution used most commonly in ANOVA. The $F_{0.05,2,26}$ is 3.3690 for a level of significance is equal to 0.05 (or 95% confidence level). As explained below, according to the F-test consideration, effective parameters have been defined for minimum shrinkage ratio in all dimensions and for minimum warpage. In Table 4, it is apparent that the F-values of injection pressure, melt temperature, and mould temperature for the dimension G were greater than $F_{0.05,2,26}$ = 3.3690. Packing pressure, packing time and cooling time were not significant for lower shrinkage in dimension G.

Cooling time was found to be the most influential parameter on the shrinkage of L, D, and d (Table 4). For different dimensions, the parameters have different effects. For the length, L, packing time and mould temperature follows cooling time. The effects from other parameters can be negligible. For the outer dimension, D, each parameter has a statistically important effect. The parameters can be listed as cooling time, injection pressure, melt temperature, packing pressure, packing time, and mould temperature. In the inner diameter (d), the mould temperature and injection pressure follow the cooling time, respectively. The injection pressure is the most effective moulding parameter on the gauge diameter (G) and thickness (t), followed by melt temperature and mould temperature for only G. It is possible to group the dimensions as length (L, D, d), width (D, d), and thickness (G, t). The results show that the shrinkage is not uniform on the part. Shrinkage ratios in length and width are similar, but different for thickness. Table 5 shows that the cooling time (24.6%) and injection pressure (22%) were found to be effective parameters on the warpage. According to ANOVA results, H_1 hypothesis was accepted and H_0 was declined for shrinkages and warpage.

Table 3. Taguchi's L27 design table and experimental data

	Pattern ABCDEF	Shrinkage on average					Warpage on average
		G	t	d	D	L	
L1	−−−−−−	7.25	5.33	3.67	1.37	0.86	1.43
L2	−−−−00	7.67	6.58	1.56	1.33	0.81	0.56
L3	−−−−++	7.33	5.79	1.47	1.11	0.59	0.24
L4	−000−−	6.88	5.50	3.94	1.66	1.15	1.29
L5	−00000	7.54	6.08	2.31	1.33	0.78	0.45
L6	−000++	7.25	5.67	1.75	0.96	0.60	0.41
L7	−+++−−	6.50	4.96	4.61	1.25	1.11	0.93
L8	−+++00	6.33	5.79	2.53	0.83	0.74	0.52
L9	−+++++	6.17	5.92	1.53	0.45	0.58	0.83
L10	0−0+−0	7.29	4.63	2.83	1.13	0.80	0.87
L11	0−0+0+	7.25	5.92	2.25	0.91	0.71	1.86
L12	0−0++−	6.83	5.92	2.67	1.09	0.73	1.12
L13	00+−−0	6.33	5.38	4.39	1.09	0.84	0.96
L14	00+−0+	5.92	5.42	2.61	0.76	0.65	0.95
L15	00+−+−	6.17	5.42	3.89	1.13	0.97	2.65
L16	0+−0−0	6.58	4.46	2.61	0.93	0.71	1.64
L17	0+−00+	5.58	4.46	1.75	0.55	0.60	0.83
L18	0+−0+−	6.25	371	2.72	1.03	0.80	1.91
L19	+−+0−+	6.54	5.08	1.97	0.80	0.71	0.47
L20	+−+00−	6.17	3.92	5.72	1.22	1.03	1.25
L21	+−+0+0	6.21	4.54	3.56	0.88	0.74	1.71
L22	+0−+−+	5.08	4.33	2.25	0.49	0.58	0.51
L23	+0−+0−	5.92	4.83	3.94	1.01	0.84	1.36
L24	+0−++0	4.63	2.96	2.42	0.54	0.64	0.99
L25	++0−−+	5.75	2.92	2.28	0.63	0.70	2.00
L26	++0−0−	6.50	3.04	5.39	1.09	1.19	3.58
L27	++0−+0	5.75	5.50	2.92	0.65	0.69	1.71

Table 4. The ANOVA results of shrinkage ratios

Source	DF	G			t			d			D			L		
		F	P	C [%]	F	P	C [%]	F	P	C [%]	F	P	C [%]	F	P	C [%]
(A) Injection Pressure	2	21.56	0.00	42	9.84	0.00	45.9	3.75	0.05	8.3	27.5	0.00	21	0.7	0.52	1.3
(B) Melt Temperature	2	12.99	0.00	25.3	2.37	0.13	11.1	0.24	0.79	0.5	18.9	0.00	14.5	0.08	0.92	0.2
(C) Mould Temperature	2	5.96	0.01	11.6	0.76	0.49	3.6	5.11	0.02	11.3	4.88	0.03	3.7	4.62	0.03	8.4
(D) Packing Pressure	2	2.16	0.15	4.2	0.22	0.80	1	0.72	0.51	1.6	10.2	0.00	7.8	1.46	0.27	2.7
(E) Packing Time	2	1.11	0.36	2.2	0.63	0.55	3	2.81	0.09	6.2	7.74	0.00	5.9	6.04	0.01	11
(F) Cooling Time	2	0.62	0.55	1.2	0.6	0.56	2.8	25.6	0.00	56.6	54.6	0.00	41.7	35.23	0.00	63.9
Residual Error	14			13.6			32.7			15.5			5.4			12.7
Total	26			100			100			100			100			100

DF: Degree of Freedom, F: F test indicator, P: Probability, C%: Contribution

Table 5. The ANOVA results of warpage

Source	DF	Warpage		
		F	P	C%
(A) Injection Pressure	2	6.0	0.01	22
(B) Melt Temperature	2	2.59	0.11	9.5
(C) Mould Temperature	2	1.78	0.21	6.5
(D) Packing Pressure	2	2.89	0.09	10.6
(E) Packing Time	2	0.29	0.75	1.1
(F) Cooling Time	2	6.69	0.01	24.6
Residual Error	14			25.7
Total	26			100

DF: Degree of Freedom, F: F test indicator, P: Probability, C%: Contribution

3.2. Shrinkage

Figure 4 shows the effects of the parameters on shrinkage in the five dimensions. The horizontal axis shows the moulding parameters and their levels. The average shrinkage ratios on the dimensions are L 0.78%, D 0.97%, d 2.95%, G 6.43%, and t 4.96%.

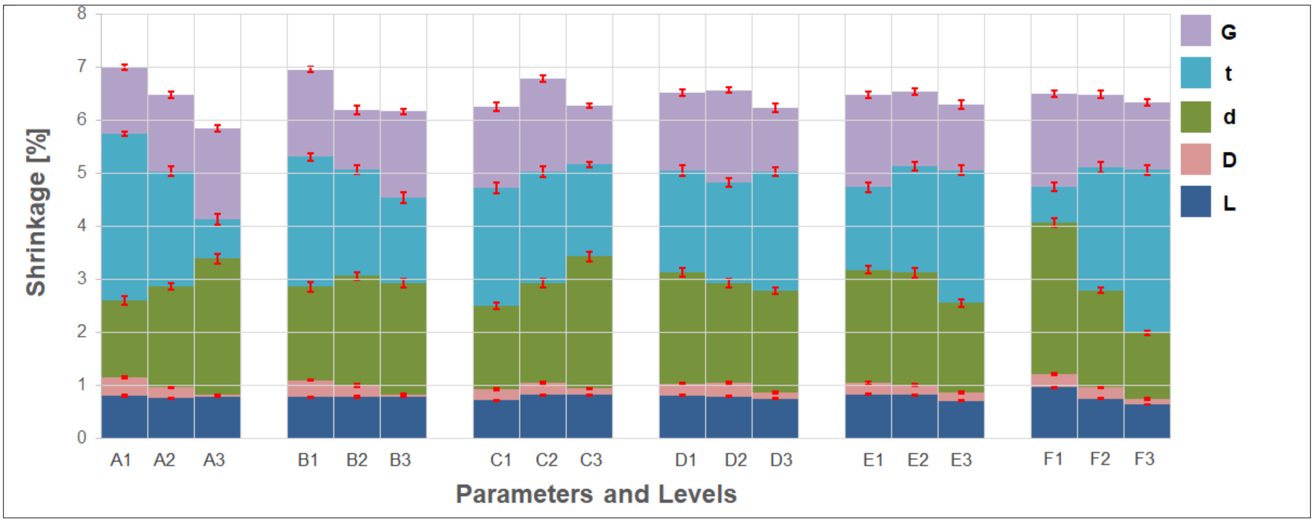

Figure 4. The main effect plots of shrinkage ratios

In the previous studies, researchers declared that the melt temperature, packing pressure, mould temperature, and injection pressure are influential on the shrinkage, form errors on the part surface, sink marks, and dimensional accuracy of the materials, which are ABS, PS, PC, HIPS, and HDPE [21-23, 40-42]. The effects of melt temperature in this study are similar to the results of Jansen et al. (1998) [23]. A similar result with a study by Kurt et al. (2009), which was performed on a dimmer cover part moulded with ABS [21], was obtained. Increasing injection pressure has made shrinkage higher on the SMPU parts, but the effects of mould temperatures on the materials were not similar in both studies. On the other hand, the effect of mould temperatures on the shrinkage of SMPU was found similar to the results of surface error of PC in the study [40]. A possible reason for that could be the high level of mould temperature, which is close to T_g (318 K) of the SMPU. Another possible reason for the effects of mould and melt temperatures on the shrinkage ratios could be the part thickness (4 mm). In this study, to determine the effect of cooling time in three different levels, which were calculated according to the specimen thickness, were applied. Higher mould and melt temperature combinations (L7-L9) shown in Table 3, shrinkage ratios in length and width were measured lower than the average at longer cooling time, but in Z as vice versa. At the end of longer cooling time and packing time, ejection temperature of the parts decreased and thus shrinkages decreased after the moulding. This result also explains how mould temperature affects the ejection temperature on the SMPU parts. It must also be highlighted that there is the influence of specimen geometry on shrinkage and on cooling time. The pins in length have restricted the shrinking of the specimen. The specimens, which were kept longer in the mould, have reached their shrinkage limit because of the mould geometry.

In the previous studies performed on various plastics, mostly packing pressure was found to be effective on shrinkage; however, for the SMPU parts, the packing time was found to be more effective than packing pressure. That might be a result of the packing pressure, which was applied in lower values as compared to the other studies, and it was changed between 20 MPa and 42 MPa, as depending on the injection pressure.

In the SMPU part, the shrinkage ratios in three main directions, which are flow direction (length), perpendicular to the flow direction (width), and through the thickness, have different values from each other. Their values are 1.5%, 2%, and 5.8% on average, respectively. Shrinkage ratios in length and in width were affected by cooling time (65.9%), mould temperature (8.9%), and packing time (7.6%). While increasing the cooling time and packing time, it was observed that the shrinkage was decreasing. It was seen that an increase in mould temperature makes the shrinkage increase. The injection pressure (55.2%) and melt temperature (18%) have been effective on the shrinkage in thickness. It was also observed that the shrinkage also decreased by increasing injection pressure and melt temperature. The reason for different shrinkage ratios in three directions is the molecular chain orientations and geometrical restrictions on the part (the hole pins). The pins cause the limitation of the shrinkage in length. The effect of mould temperature could be explained by the ejection temperature. When the mould temperature is higher, the ejection temperature will also be higher depending on packing time and cooling time. The interaction between the T_g and ejection temperature has resulted by increasing the shrinkage. The effect of injection pressure and melt temperature together can be explained by stating that easy flow provides good filling and lower shrinkage.

3.3. Warpage

Concerning warpage, it was observed that the cooling time, injection pressure or packing pressure were effective as expected [27, 28]. At medium to high levels of injection pressure, the warpage increased because of additional stress on the melted SMPU. Warpage was also reduced due to the increased cooling time and solidity. When the part was kept in the mould for a longer time, this process would provide less shrinkage and warpage. In addition, the residual stresses should be considered separately. When the injection pressure was higher, then the residual stresses in the SMPU part slightly increased [19]. Residual stresses decrease with an increase in the packing pressure. While increasing the cooling time, there is a decrease in residual stresses. This explains why warpage was going down by increasing the packing pressure. According to the literature, packing pressure mostly has the opposite effect on internal stresses and then warpage. However, for the SMPU material, this could be explained by stating that the molecular chains of SMPU need higher packing ratios than general-purpose plastics.

Figure 5 shows the mean of warpage value as 3.3 mm. As the most influential parameters, injection pressure and cooling time have caused differences in warpage values around 2.2 mm. At high injection pressures, the warpage dramatically increases. Warpage gets higher from 2 mm to 4 mm, while the injection pressure is increased. Between the lower and the medium levels, the change is sharper. This result could be explained by internal stresses, which can be rising due to the higher injection pressure and the freezing process inside the part before the relaxation. After the ejection, the part has no physical limits, so it can be relaxed by deformation via leading of the internal stresses.

The second-degree effective parameter, which is cooling time, caused the warpage to decrease from 4.6 mm to 2.4 mm, while cooling time was increasing. It shows that at the high level of cooling time, the SMPU parts have had the lowest warpage. The reason for the effect of cooling time could be related to the moulding temperatures, and especially to the mould temperature. The highest level of mould temperature (313 K) is quite close to the T_g of the SMPU (318 K); therefore, the material needs to stay longer in the mould to get more stiffness. According to ANOVA results, the best variation of moulding parameters for lower warpage can be stated as A1, B1/B2, C1, D3, E1, and F3.

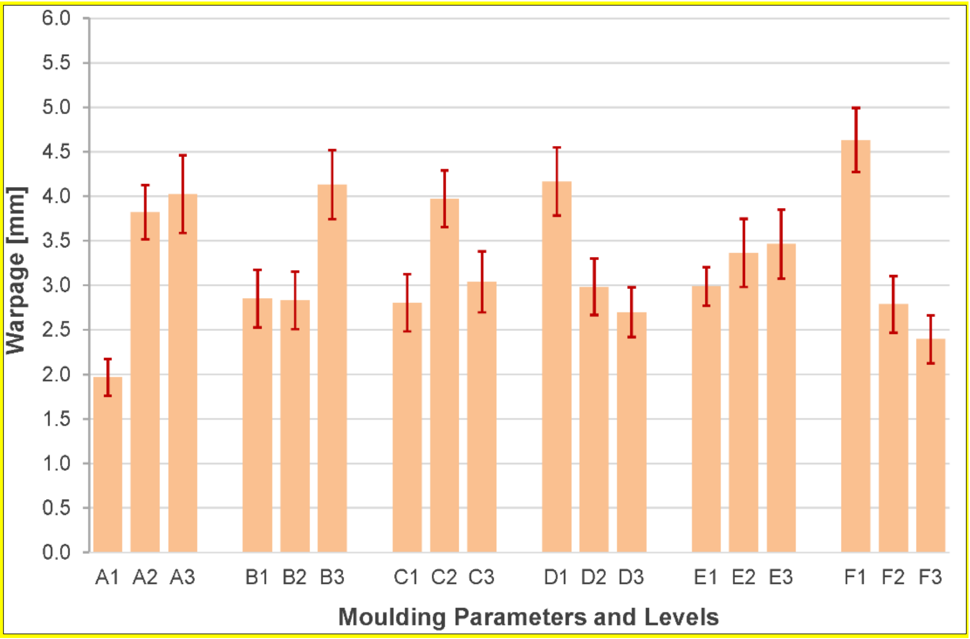

Figure 5. The main effect plots of warpage

The literature summary describes that melt and mould temperatures, packing pressure, packing and cooling times are effective factors on the warpage of parts, which were moulded with different plastic materials. However, the ANOVA results showed that only the injection pressure and the cooling time has a statistically significant effect on the warpage of SMPU. As seen in Figure 5, there is at least a 1 mm warpage difference between the levels of the parameters, so the effect of other parameters should be considered. As similar to the results of a study by Kabanemi et al. (1998), it was found that the warpage decreased by a longer cooling time in this study. Increasing of warpage with the increasing of injection pressure, showed that the shear stresses during the filling have an important impact on the SMPU tensile test bar-shaped part [26]. A similar description was used by Gao and Wang (2009) to explain the effects of packing pressure [24]. Consequently, the warpage is a result of stress relaxation, and it could happen just after moulding or after for a long time, since the warpage leads to stress-related cracking depending on the environmental conditions.

3.4. The relationship between shrinkage, warpage and residual stress

The relationship between shrinkage, warpage, and residual stresses in the specimen was investigated by Spearman's Rho Correlation analysis based on the experimental data. According to correlation analysis results in Table 6, there is a proportional relation between the shrinkages in X-axis and in Y-axis. Similarly, there is a proportional relation between the warpage and the shrinkages in X and Y-axes, (α=0.01 and α=0.05) respectively. Another property is the warpage, which has a strong relation (α=0.05) with compression residual stresses in the specimen as it was explained in [19]. It was found that there is an inverse relation between warpage and compression residual stresses. Therefore, when compression residual stresses increased, the obtained warpage on the specimen decreased. In addition, no relation has been noticed among shrinkage in thickness, warpage, and residual stresses. In Figure 6, the effects of moulding parameters were compared to each other in a pie diagram for five different dimensions and warpage. In the figure, *L, t, d, D, G* represents five different dimensions as explained in subsection 3.2, and *W* represents the warpage.

Table 6. Summarises the effects of moulding parameters on shrinkage and warpage

	Shrinkage X [%]	Shrinkage Y [%]	Shrinkage Z [%]	Warpage [mm]	Compression Residual Stress [MPa]	Tensile Residual Stress [MPa]
Shrinkage X [%]	1					
Shrinkage Y [%]	0.999**	1				
Shrinkage Z [%]	-0.149	-0.167	1			
Warpage [mm]	0.494**	0.486*	-0.342	1		
Compression Residual Stress [MPa]	-0.355	-0.348	0.481*	-0.458*	1	
Tensile Residual Stress [MPa]	-0.074	-0.068	0.002	0.099	0.304	1

** Correlation is significant at the 0.01 level (2-tailed), *. Correlation is significant at the 0.05 level (2-tailed), c. Listwise N=27

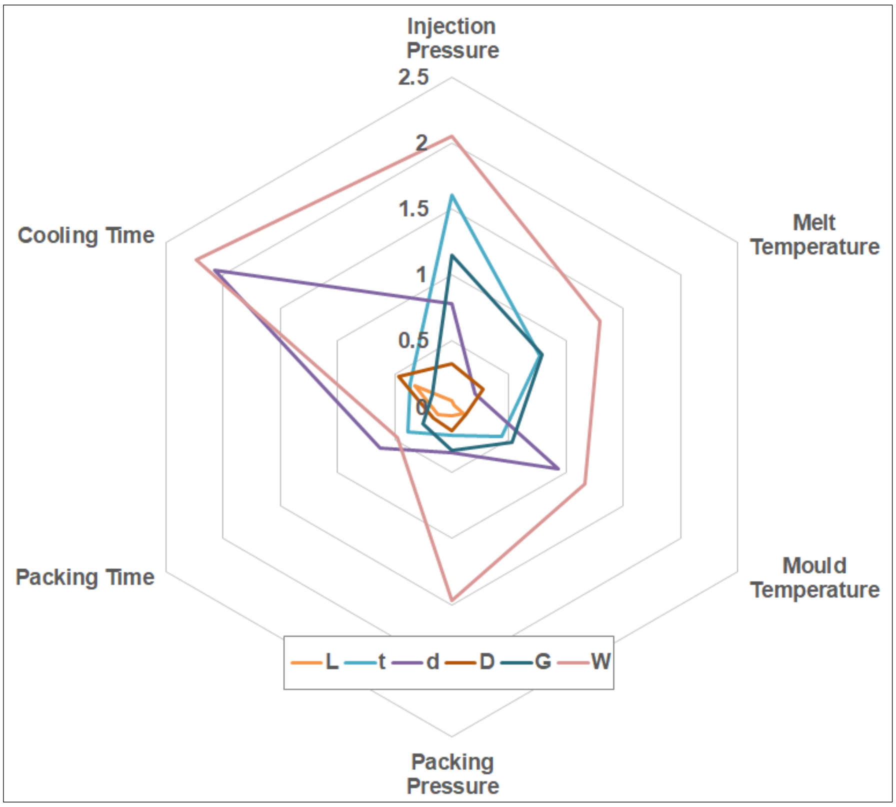

Figure 6. Comparison of parameters effects for shrinkage and warpage

4. Conclusions

In this study, the effects of injection moulding parameters on the shrinkage and warpage of shape memory polyurethane (SMPU) parts are presented. The study results show that the injection moulding parameters have significant effects on the shrinkage and warpage of temperature-sensitive thermoplastic SMPU parts. The main results obtained in this study.

Shrinkage ratios in three directions, which are along the flow direction (in length), perpendicular to the flow direction (in width), and through the thickness, are different from each other.

While cooling time, mould temperature, and packing time have effects on a long and perpendicular line to the flow direction, injection pressure and melt temperature have effects on thickness. In addition, the shrinkage ratios along the flow direction and perpendicular to the flow direction, were found to be lower than the shrinkage ratio for thickness.

Cooling time is the most effective process parameter on shrinkage in length, width, and warpage, since it also affects the ejection temperature of moulded parts.

Ejection temperature for SMPU is more important than general-purpose plastics because of its T_g shape-memory activation temperature. When the ejection temperature of the moulded part was higher than the T_g, it led to an uncontrollable permanent shape of the part to be formed.

The injection pressure was found to be the second effective parameter on shrinkage, thickness, and warpage. The part geometry is also an important factor to restrict the shrinkage of the SMPU parts.

Acknowledgments: Sukran Katmer thanks to the Scientific and Technological Research Council of Turkey (TUBITAK) for the provided PhD scholarship (BIDEB-2214/A) for this study.

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Manuscript received: 28.10.2020