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Influence of Process Parameters on the Morphologies of Micro-Injection Molded Polyformaldehyde Parts

The morphologies of micro-injection molded parts are influenced by the process parameters. In this paper, the influence of injection speed, mold temperature and melt temperature on the morphologies of micro-injection molded polyformaldehyde (POM) parts with different thicknesses were investigated by a single factor experimental method; the morphological structure of the parts was characterized by polarized light microscopy. The scale effect on the crystallization behavior and internal morphology of micro-injection POM parts was analyzed. The results indicated that the scale effect had a great influence on the hierarchical morphology in the thickness direction of the parts. The micro-parts with a thickness of 1.0 mm showed a skin-core structure including the skin layer, fine grain layer, oblate spherulite, and spherulite core layer, and the micro-parts with a thickness of 0.2 mm showed a skin-core structure with the skin layer, fine grain layer, and the spherulite core layer, and a larger thickness ratio of the spherulite core layer. As injection speed, mold temperature and melt temperature increase, the fine grain layer gradually disappears and the size of core spherulite tends to become larger, the thicknesses of the skin layer of all the micro-parts decrease and that of the 0.2 mm micro-parts decreases significantly.

1 Introduction

The process parameters during micro-injection molding may lead to special morphology, structure, and performance of micro-parts, which is different from conventional injection molding (Selada et al., 2011; Nian et al., 2005). It was proposed that the morphology of isotactic polypropylene parts presented three layer structure including highly oriented non-spherulitic layer, transition layer, and a spherulite core layer. Also, it was pointed out that the orientation and the thickness of the oriented skin layer were a function of the melt tempera-

ture, and as the melt temperature increases, the thickness of the skin layer tends to decrease (Kantz et al., 1972; Fitchmun and Mencik, 1973). The molecular orientation in injection molding of acetal homopolymer was studied, and the overall structure of the parts includes three layers: skin, "transcrystalline region", core layer (Clark, 1967), Bowman (1981) divided acetal copolymer into five layers, gathered into three zones: skin, shear zone, core. The outer three layers possessed significant preferred chain-axis orientation in the crystalline phase, while the two layers at the center of the molding were equiaxed (Bowman, 1981). The morphology of polypropylene parts can be divided into five layers in the thickness direction. The dominant factors affecting the thickness of skin layer, the content of β -crystal at the junction of skin and core layer, and the average size of the spherulite are melt temperature and injection speed (Kamal et al., 2017). It was found that when the mold temperature increased from 60°C to 120 °C, the size of the β-crystals in isotactic polypropylene becomes larger; high injection speeds can promote the formation of the β-crystal, but the value of the injection speed must be within a certain range (Chen et al., 2016). Sun et al. (2017) investigated the morphological evolution and the β -crystals distribution of isotactic polypropylene with the assistance of a long chain branched structure under micro-injection molding condition, and found that the introduction of branched polypropylene can improve the orientation degree of the molecular chains and decrease the thickness of the core layer, thereby inevitably suppressing the "skin-core" structure. By comparing the morphology of micro-injection parts and traditional injection molded parts, it was found that the traditional parts exhibited a skin-core structure, and the micro-parts exhibited a special no core structure (Giboz et al., 2009; Zhang et al., 2008; Kamal et al., 2010). Liu et al. (2012) pointed out that the micro-injection molded parts showed the similar skin-core structure in the thickness direction of the traditional injection molded parts, but there is a greater proportion of the skin layer. The shear layer of the parts showed a shish-kebab structure that is highly oriented in the direction of flow, and the micro-injection molded parts had a more even distribution of lamellae thickness (Liu et al., 2012). The morphological results showed that the thickness of the skin layer and fine grain region were significantly affected by the injection speed

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and mold temperature, and the thickness of the spherulite layer was mainly determined by the mold temperature (Zhang et al., 2015). It was found that micro-injection molded parts exhibited a shish-kebab structure which was highly oriented along the flow direction; the injection speed and mold temperature played an important role in the crystallization behavior of micro-injection molded POM parts, and the increase of mold temperature was beneficial to the integrity of the crystal structure (Baldi et al., 2014). In the investigation of the morphological structure of micro-injection molded polypropylene parts, Li et al. (2013) pointed out that the morphology of 1.0 mm parts showed a typical skin-core structure, while 0.2 mm micro-parts presented non-frozen layer and nonspherulite structure, almost entirely shear layer with shish-kebab structure. The proportion of each layer changes along the flow direction, while the injection speed had the greatest effect on the morphological structure of the parts, followed by the mold temperature and the melt temperature (Wang et al., 2017). Therefore, it is very important to understand the mechanism of the scale effect and processing parameters on the morphology and structure of the micro parts.

In this paper, thin-walled parts were molded by a commercial injection machine with crystalline POM under different process conditions (Wang et al., 2013). Influence of process parameters (mold temperature, injection speed and melt temperature) on morphology of the micro-parts with the thickness of 1.0 mm and 0.2 mm were investigated by a single factor experimental method. Morphologies of the micro-parts were characterized by means of PLM, and the influence of processing parameters and scale effects on crystallization and morphology of micro-injection molded POM parts were investigated. The purpose of this work is to analyze the relation between processing parameters and morphological structure of POM micro-parts, and the mechanism of the processing parameters on the morphology and structure. It is of great importance to control the morphological structure and improve the mechanical properties of micro-injection molded parts.

2 Experimental Part

2.1 Materials

The material used in this work is a commercially available copolymer polyformaldehyde (cPOM, M90-44) from Poly Plastics Co., Tokyo, Japan. The value of the melt flow rate at standard testing conditions (190 °C, ISO 1133) is 9 g/10 min, the specific gravity is 1.41, and the flexural modulus, flexural strength and tensile strength at standard testing conditions are 2580 MPa, 90 MPa, and 60 MPa respectively.

2.2 Micro-Parts and Mold Design

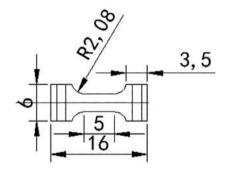
According to the ASTM standard, the micro-parts were designed as dumbbell shaped micro tensile samples. The geometry of the micro sample is shown in Fig. 1, and the thickness of the parts are 1.0 mm and 0.2 mm respectively. The mold was designed as a set of changeable mold inserts with two-cav-

ities, and the thickness of the cavity is 1.0 mm and 0.2 mm respectively.

2.3 Sample Preparation and Characterization Methods

2.3.1 Micro-Injection Molding

The single factor experimental method was used to study the influence of process parameters (injection speed, melt temperature, and mold temperature) on the morphological structure of the micro-injection molded POM parts with different thicknesses. The process setups are shown in Table 1, the baseline of the single factor experiments is a melt temperature of 210 °C, a mold temperature of 110 °C and an injection speed of 220 mm/s. The experiments were carried out using a Victory-28 CC-200 injection molding machine (Engel, Schwertberg, Austria) with a screw diameter of 18 mm, the maximum injection speed of 600 mm/s, and the maximum injection pressure of 220 MPa. The mold temperature controller used in the experiment was a GMC serial oil circulation control system, produced by Zhangjiagang Great Wall Matsui Machinery Co. Ltd., Zhangjiagang, PRC, with the temperature range of 60 to 160 °C, temperature control accuracy of ±1 °C.



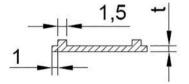


Fig. 1. Geometry size of the micro-sample

Part thickness	0.2 mm, 1.0 mm
Injection speed (mm/s) Mold temperature (°C) Melt temperature (°C)	40, 100, 160, 220, 280, 400 80, 90, 100, 110, 120 190, 200, 210

Table. 1. Process setups for injection molding of the micro-parts with the thickness of 0.2 mm and 1.0 mm

2.3.2 Sampling Method

A slice was cut along the longitudinal symmetry section of the parts (parallel to the plane of thickness direction (ND)-flow direction (FD)) using the rotary microtome (model RM2235, Leica, Wetzlar, Germany) according to the sample cutting method shown in Fig. 2; the thickness of the micro-slices is 8 µm. The slice was placed on an alcohol-treated slide and then covered with a coverslip to observe the morphological structure with PLM. Three points A, B, and C were marked for polarized observation. Morphological changes along the flow di-

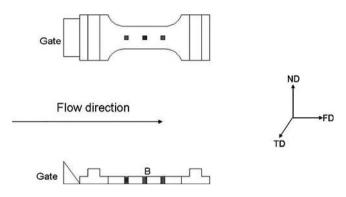


Fig. 2. Sample preparation methods and processes

rection can be characterized by morphologies at different positions along the flow direction.

2.3.3 Polarized Observation

The samples were fixed on the stage of a PLM, then the morphologies of micro-parts were analyzed and the effects of process parameters and scale effect on the crystallization behavior and internal morphologies of the POM micro-parts were investigated. The PLM (model BX61, Olympus, Tokyo, Japan) with an eyepiece of 10x magnification and an objective lens of 5, 10, 20 and 50x magnifications were adopted in the experiments.

3 Results and Discussion

3.1 Morphological Structure of the Micro-Injection Parts

Figure 3 shows the PLM images at point B of the POM microparts with the thickness of 1.0 mm and 0.2 mm molded under at the injection speed of 220 mm/s, melt temperature of $210 \,^{\circ}\text{C}$, and mold temperature of $110 \,^{\circ}\text{C}$.

The results show that all the micro-parts presented a skincore structure Fig. 3A shows that the morphologies of the 1.0 mm injection molded parts present a skin-core structure in-

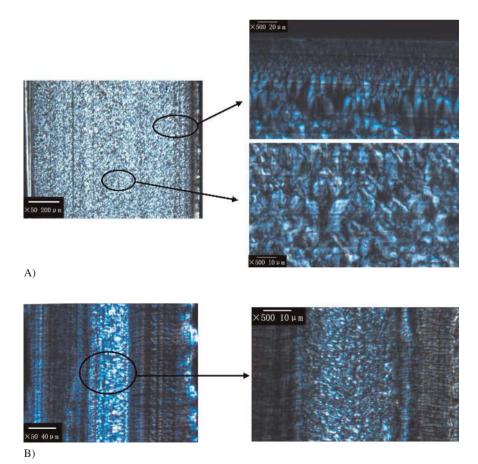


Fig. 3. PLM images of POM micro-parts with a thickness of 1.0 mm (A) and 0.2 mm (B) at point B

cluding the skin layer, transcrystaline layer with twisted lamellae, large oblate spherulitic layer, and the spherulitic core layer, as shown in Fig. 4. The skin layer consists of the frozen layer and a highly oriented shear layer and accounts for a small proportion. A smaller size of the fine grain layer appeared occasionally within the matrix of the twisted lamellae structure which refers to a model by Clark showing lamellae growing perpendicular to the surfaces with twisting (Clark, 1967; 1973); the oblate spherulitic layer consists of quasi-parabolic spherulites, which are the signature of a temperature gradient, and the core layer has random spherulites. Different from the morphology of traditional injection molding, there is almost no transitional layer visible in the skin layer, only the frozen layer, and the shear layer. The transition layer between the skin layer and oblate spherulitic layer is a fine spherulitic layer, in which spherulites are fine and symmetrical. The size of the quasi-parabolic spherulites in the oblate spherulitic layer is lager. The results in Fig. 3B show that the morphologies of the 0.2 mm micro parts presented the skin-core structure including the skin layer, the transition layer composed of the fine grains, and the spherulite core layer. The skin layer consists of the frozen layer and a highly oriented shear layer. The transition layer is mostly composed of the fine grain layer. The proportions of the skin layer and transition layer of 0.2 mm micro-parts were larger than those of 1.0 mm micro-parts. The random spherulites were distributed almost in the spherulite core layer, rather than near the wall as for the 1.0 mm part.

The formation mechanisms of the distinctive layered structure are different from the traditional injection molded parts during the micro-parts molding process, and it can be explained by the flow behavior and temperature gradient of the polymer melt in the microcavity. In the melt filling stage, when the high-temperature melt entered into the low-temperature cavity under the driving of high pressure, the hot melt contacted the cold mold wall, some of the random molecular chains adhered to the lowtemperature mold wall and solidified instantly with little crystallization, thus forming the frozen layer in the surface of the part. While the nearby melt continued to flow, the molecular chains stretched and oriented along the flow direction, thus forming the shear layer. Since the mold wall temperature is much lower than the melt temperature, the nucleation rate of the melt next to the skin layer is very high at a large temperature gradient. Due to the high cooling rate, the high nucleation density results in the small spherulites. In the late stage of forming, the melt crystallization is controlled by the nucleation stage; the nucleation rate near the skin layer is high enough to form lots of nuclei, the growth of crystal outward and axial is limited. The enhance-

ment of the molecular thermal motion of the high-temperature melt near the core leads to no spontaneous nucleation, which results in sufficient growth space for the fine spherulitic crystals to grow. Due to the uneven distribution of heat, the melt in the outer region near the mold wall always has a greater cooling rate than the melt in the inter-region, the oblate spherulites being the result of the competition between the nucleation rate and the crystal growth rate. As the temperature increases, the nucleation rate decreases sharply, and the crystal grows more freely because of less hindrance due to other lamellae, resulting in the over-growth of the crystals and forming the oblate spherulites which are actually quasi-parabolic spherulites (Lovinger and Gryte, 1976; Piorkowska et al., 2004). Finally, the melt in the core layer has sufficient time and space for the crystals to grow due to higher melt temperatures and lower shear effects, the oriented molecular chains have sufficient time to relax, the orientation state cannot be maintained to form an isotropic spherulitic structure.

For the micro-parts with a thickness of 0.2 mm, the heat exchange with the mold wall is enhanced, the melt in the cavity is easier to cool down and the oriented molecular chains are frozen, so the proportion of skin layer of 0.2 mm parts is much larger than that of 1.0 mm micro-parts; on the other hand, since the heat loss from the mold wall to the core layer is larger than that of 1.0 mm parts, the transition layer inclines to the core, and the large spherulites appear at the higher temperature region in the core layer.

3.2 Effect of Injection Speed on Morphological Structure of POM Micro-Injection Molded Parts

Figure 5 contains the PLM images of the POM micro-parts with the thickness of 1.0 mm and 0.2 mm under different injection speeds. The results show that all the micro-parts presented a skin-core structure. The results in Fig. 5A show that with the increase of injection speed, the fine grain layer of the micro-parts with a thickness of 0.2 mm tends to disappear gradually, the size of the oblate spherulites adjacent to the small grain layer increases, and the core layer consists of random spherulites. The results in Fig. 5B show that with the increase of injection speed, the thickness of the core layer of the micro-parts with a thickness of 0.2 mm increases gradually; when the injection speed exceeds 280 mm/s, the fine grain layer gradually disappears, and the core layer of the micro-parts consists entirely of randomly oriented spherulites.

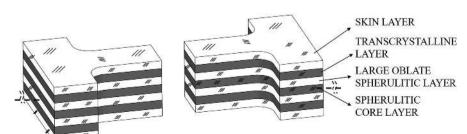
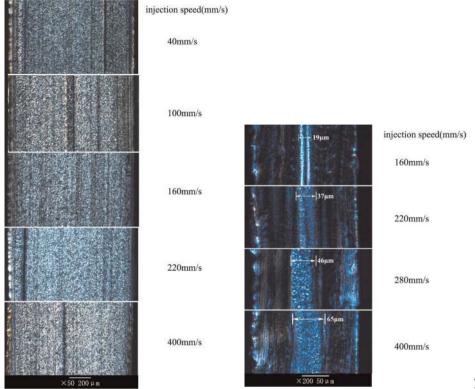


Fig. 4. The skin-core structure with a thickness of 1.0 mm



B)

Fig. 5. PLM images of micro-parts with the thickness of 1.0 mm (A) and 0.2 mm (B) molded under different injection speeds

With increasing injection speed, the shear effect of the melt increases, resulting in a higher melt temperature due to a large amount of dissipated heat. The growth rate of the crystal is greater than the rate of nucleation at the high melt temperature and tends to form larger spherulites. Thus, the spherulite size in the core layer of the micro-parts with a thickness of 0.2 mm increases with increasing injection speed.

A)

The results in Fig. 6 indicate that the thickness of the skin layer of 1.0 mm and 0.2 mm micro-parts decrease with increasing injection speed, and the skin layer occupies a larger proportion in the thickness direction of 0.2 mm parts. In the range of the investigated injection speeds, with the increase of injection speed the enhanced shear behavior produces two effects: on the one hand, the increasing shear effect can enhance the orientation of the molecular chains and cause a thicker skin layer; on the other hand, the increasing shear effect produces a large amount of heat, which causes the melt temperature to rise. The increase of the melt temperature makes the oriented molecular chains having enough time to relax and causes the skin layer of the parts to decrease. When the injection speed increases to a certain value, the phenomenon of wall slip occurs and reduces the shear effect while the contact thermal boundary under the adhesion boundary becomes incomplete, which affects the cooling effect of the mold wall on the melt. The comprehensive effect causes the thickness of the skin layer to decrease with increasing injection speed.

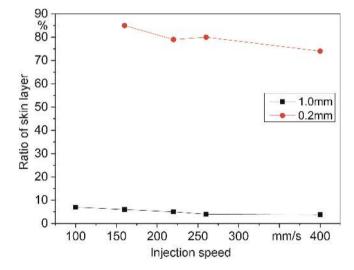


Fig. 6. Relation between the ratio of the skin layer of micro-parts and injection speed for 1.0 mm and 0.2 mm parts

3.3 Effect of Melt Temperature on Morphological Structure of POM Micro-Injection Parts

Figure 7 presents PLM images of the micro-parts with the thickness of 1.0 mm and 0.2 mm molded under different melt temperatures. The results in Fig. 7A indicate that with the increase of the melt temperature, the fine grain layer tends to dis-

appear gradually; the size of the oblate spherulites adjacent to the fine grain layer becomes larger. For the 0.2 mm parts, as the melt temperature increases, the thickness of the skin layer decreases, and the crystal size of the core layer increases, as shown in Fig. 7B.

Upon increasing the melt temperature, the viscosity decreases, the flowability increases, which results in shorter shear time and a lower orientation degree of the macromolecular chains. Meanwhile, high temperature makes the oriented macromolecular chains having enough time to relax, therefore the two effects cause the shear layer thickness to decrease. The relationship between the thickness of the skin layer and the melt temperature is shown in Fig. 7. As the melt temperature increases, the nucleation rate decreases and the crystals grow more freely because of less hindrance due to other lamellae. The melt tends to produce larger size spherulites, therefore the proportion of the fine grain layer gradually decreases.

Figure 8 shows the ratio of the skin layer of the micro-parts with the thickness of 1.0 mm and 0.2 mm molded under different melt temperatures. The results indicate that the fraction of

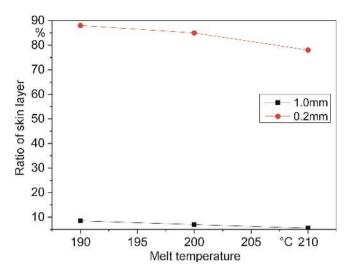


Fig. 8. The ratio of the skin layer of micro-parts molded under different melt temperatures (1.0 mm and 0.2 mm)

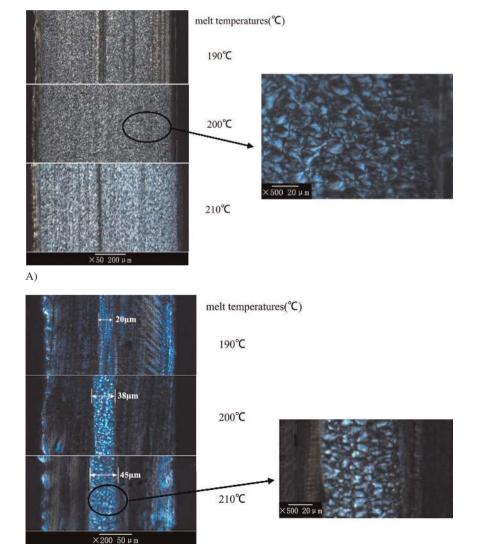
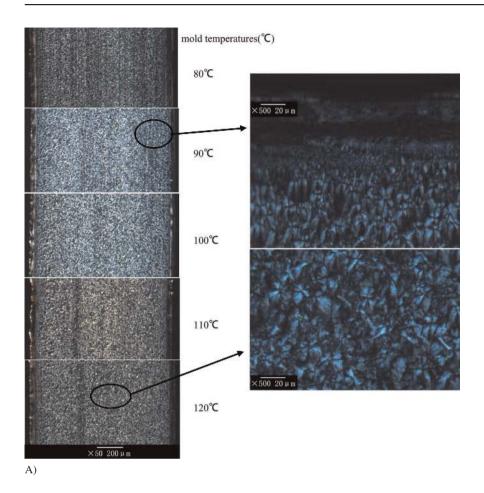


Fig. 7. PLM images of micro-parts with the thickness of 1.0 mm (A) and 0.2 mm (B) molded under different melt temperatures



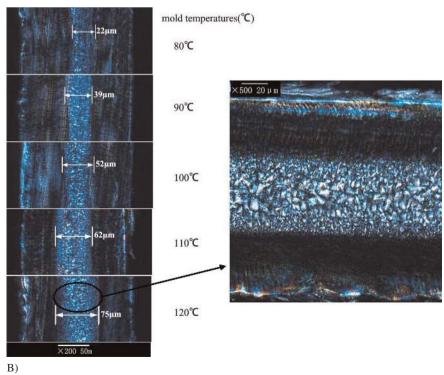


Fig. 9. PLM images of micro-parts with the thickness of 1.0 mm (A) and 0.2 mm (B) molded under different mold temperatures

the skin layer of the micro-parts with a thickness of $0.2~\mathrm{mm}$ is much larger than that of $1.0~\mathrm{mm}$ parts. Moreover, with increasing melt temperature, the ratios of the skin layer of both $1.0~\mathrm{mm}$ and $0.2~\mathrm{mm}$ parts show a decreasing trend, and the ratio of $0.2~\mathrm{mm}$ parts changes more obviously than that of $1.0~\mathrm{mm}$ parts. The results indicate that the impact of increasing melt temperature on $0.2~\mathrm{mm}$ micro-parts is greater than the impact on the micro-parts with a thickness of $1.0~\mathrm{mm}$.

The reduction of the thickness of the parts causes the cooling rate of the melt to increase, and thus the thickness of the skin layer of the thinner parts is relatively larger. The size of the spherulites in the core layer increases from the outer side to the center of the spherulites core layer because of the decrease of the cooling rate, and the change of the size of the spherulites is the result of the combined effect of shear and heat distribution. Firstly, the portion of the melt near the skin layer is rapidly cooled down to form the small size spherulites, while the melt under a relatively slow cooling rate tends to form the larger size spherulites. Secondly, the spherulites near the skin layer may be formed during the filling and holding stages, and higher melt pressure and higher shear stress promote the rate of nucleation, while the crystals do not have enough space to grow. Thirdly, the melt in the center may cool down in the cooling stage, due to higher melt temperature, lower pressure and shear stress, the melt at the center of the core layer is conducive to grow rapidly, thus forming the larger size spheru-

3.4 Effect of Mold Temperature on Morphological Structure of POM Micro-Injection Molded Parts

Figure 9 presents the PLM images of the micro-parts with a thickness of 1.0 mm and 0.2 mm molded under different mold temperatures. It is shown that with the increase of mold temperature, the thickness of the skin layer decreases, the fine grain layer tends to disappear gradually, the size of the oblate spherulite adjacent to the small grain layer increases, and the thickness of the spherulites core layer increases. Figure 9B also shows that, with increasing mold temperature, the thickness of the fine grain layer decreases and the size of core spherulites increases.

Figure 10 shows the ratio of the skin layer of the micro-parts with the thickness of 1.0 mm and 0.2 mm molded under different mold temperatures. It can be seen from Fig. 10 that the proportion of the skin layer of 0.2 mm micro-parts is larger than that of 1.0 mm parts. With increasing mold temperature, the ratios of the skin layer of both 1.0 mm and 0.2 mm parts show a decreasing trend, and the ratio of 0.2 mm micro-parts varies more obviously than that of 1.0 mm parts; the influence of increasing mold temperature on 0.2 mm micro-parts is higher than that for the micro-parts with a thickness of 1.0 mm.

The mold temperature is a direct factor of heat exchange between the melt and the mold and has a significant effect on the flow behavior of the melt. With the increase of mold temperature, the difference between the melt temperature and the mold temperature decreases, and the cooling effect of the mold on the melt temperature decreases, so that the melt temperature is relatively increased. The increase of the melt temperature makes the oriented molecular chains having enough time to re-

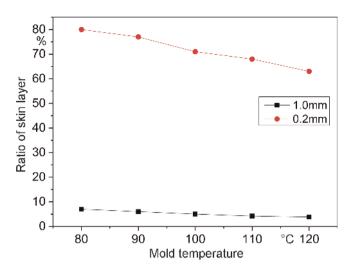


Fig. 10. The ratio of the skin layer of micro-parts molded under different mold temperatures (1.0 mm and 0.2 mm)

lax and causes the skin layer of the parts to decrease, thus the thickness of the skin layer decreases with increasing mold temperature.

4 Conclusions

It was found that the scale has a great influence on the hierarchical morphology in the thickness direction of the parts. The micro-parts with a thickness of 1.0 mm show the skin-core structure including the skin layer, fine grain layer, oblate spherulite layer, and the spherulite core layer, while the 0.2 mm microparts show the skin-core structure with the skin layer, fine grain layer, and the spherulite core layer, and a larger size of the core spherulites. As injection speed, mold temperature and melt temperature increase, the fine grain layer gradually disappears and the size of core spherulites tends to become larger, the thickness of the skin layer of all the micro-parts decreases and that of the micro-parts with a thickness of 0.2 mm decreases significantly.

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