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ORIGINAL ARTICLE

Process and properties of micro-ultrasonic powder molding with polypropylene

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Abstract Aimed at microplastic parts molding, we use a novel method of micro-ultrasonic powder molding (micro-UPM) on polypropylene (PP) microplastic parts and investigate the experimental process in detail. Firstly, the experimental results show that the temperature is maximum on the top outer edges of plastic parts and minimum on the bottom center. Then, the effects of experimental process parameters on plastic flashes are studied. The results show that under the same pressure, the flash thickness gradually thins with increased ultrasonic time. The ultrasonic time for the flashes to be automatically separated from the matrix of microplastic parts is obviously shortens with increased pressure. Finally, the tests on thermal properties, morphology, and mechanical properties of microplastic parts are also conducted. The results show that the melting point and crystallinity of microplastic parts produced by micro-UPM are higher than those of raw materials. The organizational structure shows no obvious "skin–core" structure, and its crystal form is α crystal form, which is the most common type of polypropylene. With increased ultrasonic time, the tensile strength and elongation at break of samples both have a trend of rise followed by drop before and after annealing. An ultrasonic time that is too long can lead to the degradation of materials.

Keywords Micromolding · Skin–core structure · Ultrasonic · Polypropylene powder

1 Introduction

With the development of micro-electromechanical systems (MEMS), microplastic parts have been widely used in the field of aerospace, electronic communications, and biomedical [1, 2]. Microplastic parts are small and have microsize effects in the molding process so they require high-quality molding of polymer melt. Currently, the existing micromolding technology of polymer materials mainly includes micro-injection molding and micro-hot embossing [3–7]. However, there are many challenges in the development of micromolding technologies: some polymer materials (such as ultrahigh molecular weight polyethylene (UHMWPE) and polytetrafluoroethylene) are characterized by high viscosity, poor fluidity, and difficult processing, which limit their utilization to some degree [8]. In the micro-injection molding technology, problems such as slow melt filling flow and incomplete filling of microplastic parts are encountered [9]. The mold needs to be heated in the micro-hot embossing process, the molding period is long, and the products are hard to demould. Therefore, researchers constantly explore new polymer processing technologies.

Fairbanks [10] used ultrasonic to compress molding plastic powder and found that the power particles can be melted without the addition of external heat, and the cylindrical plastic parts were prepared successfully. Paul et al. [11] optimized the strength of plastic parts by compression molding on polypropylene (PP) powder using ultrasonic welding equipment. Using polypropylene as raw material, Xie et al. [12] explored the influence of ultrasonic vibration on weld lines by installing an ultrasonic generator on the micro-injection mold. Michaeli et al. [13] developed one set of ultrasonic injection molding system. In the process, the mold temperature is kept at 80 °C through an electric heating method. The PP particles are precharged into the charging barrel. A sonotrode is used to ultrasonically vibrate the PP particles so that they are completely plasticized. The plasticized melt enters the microcavity along a side runner to completely mold microgear plastic parts. Jiang et al. [14] studied the experimental of polymer ultrasonic plastification with a homemade ultrasonic melting plastification experimental device. The results show

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that compared with heating plastification, the ultrasonic plastification is more suitable for micro-injection molding with a better plastification quality. Mekaru et al. [15, 16] applied ultrasonic waves to micro-hot embossing. The microstructure is imprinted on the surface heating plastic workpiece by vibrating the head of ultrasonic tool with microstructure. The results show that the flow property of the polymer is markedly improved under ultrasonic vibration, and the microproducts have a high accuracy. Li et al. [17] applied the ultrasonic vibration to investigate UHMWPE extrusion molding. The results show that ultrasonic vibration can significantly decrease the die pressure and apparent viscosity of the melt, thereby increasing the output of extrudate.

Based on the above studies, a micro-ultrasonic powder molding method (micro-UPM) was proposed in the research [18]. Under a certain sonotrode pressure, the PP materials are melted by heat derived from strong friction and compression among PP powder particles by ultrasonic vibration. The polymer melt is then directly molded in the microcavity. This is a very significant approach wherein polymer powder can be directly plasticized and molded in the microcavity to avoid the formation of weld marks. Compared with injection molding, the mold is simple and does not require runner systems and expensive injection molding machine equipment. In addition, the molding cycle is short, making it an effective way of saving energy and reduce costs. The thermal properties, morphology, and mechanical properties of the microplastic parts are investigated. The reported results of the research are of benefit to broaden the ultrasonic application in the polymer micromolding field and provide a new train of thought for polymer micromolding technology.

2 Experimental

2.1 Micro-UPM process

The micro-UPM process involves the following three steps:

1. Mold assembly and location. Firstly, the microcavity insert is placed on the microcavity backing plate (Fig. 1). The mold pressing plate is then placed on the mold backing plate, and the microcavity insert is compacted. The combined mold is fixed on an ultrasonic plasticizing and molding experimental platform (Fig. 2) with fastening plates. Meanwhile, the sonotrode is aligned with the charging barrel hole.
2. Powder filling and compaction. PP powder, which is a little heavier than the microplastic part, is filled into the mold cavity and the charging barrel, under the condition of no starting ultrasonic vibration, the sonotrode repeatedly moves up and down until the PP powder in the cavity is fully compacted.
3. Ultrasonic plasticization and molding. Parameters including ultrasonic power, ultrasonic time, pressure (sonotrode back pressure), and holding time are set on the ultrasonic plasticizing and molding experimental platform, after which the ultrasonic vibration is applied. The microplastic parts are completely molded, the mold is separated, the microplastic parts are taken out, and the overflowing materials and the flashes (Fig. 5b) are stripped.

The presence of polymer flashes can also avoid direct collision between sonotrode and mold so that the mold is well protected. Therefore, the role of flashes in micro-UPM is similar to those in metal hot forging. The following set of optimum process parameters were used in the molding experiment: pressure of 0.1 MPa, ultrasonic time of 4.0 s, and holding time of 5 s. The resulting microplastic gears are shown in Fig. 3. The following parameters for the gears are used: 8 teeth, 4.8 mm pitch diameter, and 20° pressure angle. The plastic parts are clear, transparent, and high quality, thereby verifying the process feasibility.

2.2 Materials and equipment

The material used in the experiment was isotactic polypropylene (iPP) powder (Sinopec Maoming Petrochemical Co., Ltd.; grade, 405). It has a density of 0.90 g/cm^3 at 25 °C. The powder particles are approximately 350 μm in size. An ultrasonic welder (China Shenzhen Hongri Ultrasonic Equipment Co., Ltd., RS2016) was used as the ultrasonic plasticizing and molding experimental platform. The experiment power was 2,600 W and the output frequency of the sonotrode was

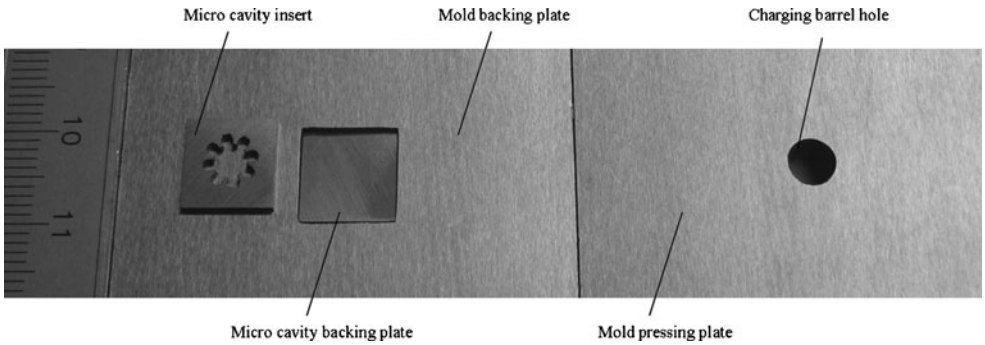

Fig. 1 Photo of combined mold

Fig. 2 Ultrasonic plasticizing and molding experimental platform

20 kHz. Differential scanning calorimeter (USA TA Instruments Co., Ltd., Q200) was used to measure the melting temperature and the melt enthalpy of various samples. The samples were heated under nitrogen atmosphere form 25 to 250 °C at a rate of 10 °C/min. The crystallinity (X_c) of various samples is calculated according to the following expression:

$$X_c = \frac{\nabla H}{\nabla H_f^0} \times 100\% \quad (1)$$

where ∇H is the melt enthalpy of various samples measured by TA Universal Analysis software and ∇H_f^0 is the melt enthalpy of the 100 % crystal of PP (209 J/g) [19]. Microplastic parts were cut to slices of 50 μm in thickness along the filling direction used a microtome (German Leica Co., Ltd., RM2135). The slices were then placed on a polarized light microscope (China Shanghai Optical Instrument Co., Ltd., XPV-400E) to examine the morphology of microplastic parts. The phase composition and orientation of microplastic parts were analyzed using a single crystal X-ray diffractometer (Japan RIGAKU Co., Ltd., R-AXIS RAPID). Tensile tests were conducted on a universal testing machine (German Zwick/Roell Co., Ltd., Z0.5). The samples were 1 mm thick, 4 mm long, and had 20 mm/min tensile displacement velocity.

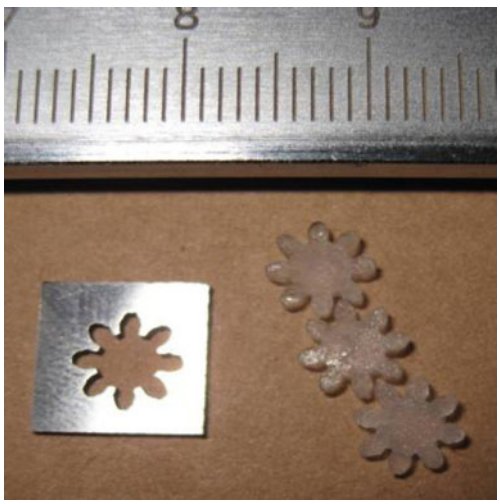

Fig. 3 Micro-UPM plastic gears

3 Results and discussion

3.1 Temperature field

Previous studies have shown that when particles are too large or the ultrasonic energy is insufficient, the heat derived from the friction and compression among particles by ultrasonic vibration is insufficient to plasticize the entire particle such that only the peripheral part of particles can be plasticized. Thus, the inner of plastic part is basically composed of recrystallized region and nonrecrystallized region (Fig. 4). The ultrasonic vibration causes friction between the mold cavity and polymer particles, which is usually more violent on the side edges of microplastic parts. The polymer degrades when the friction is too intense. Consequently, an obtained representative diagram for temperature distribution along the horizontal direction is shown in Fig. 5. The distribution of temperature assumes a wave shape, wherein the peak values of temperature in the boundary position among particles are formed, and they are beyond the melting point of polymer material. The temperature at the position of the edge in contact with the mold cavity may be higher and can even exceed the degradation temperature when it is too high, thereby resulting in a degradation (black) region. On the other hand, the height direction has a similar wavy distribution and the temperature on the contact surface of the sonotrode is the highest. Therefore, considering the comprehensive effects of temperature changes in the two directions, the top of lateral edges with the highest temperature is most likely to degrade. The bottom of central region with the lowest temperature is most likely to plasticize insufficiently. For example, the microplastic gears

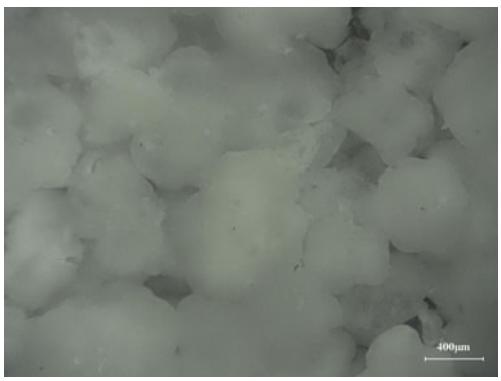

Fig. 4 Sample of insufficient plastication

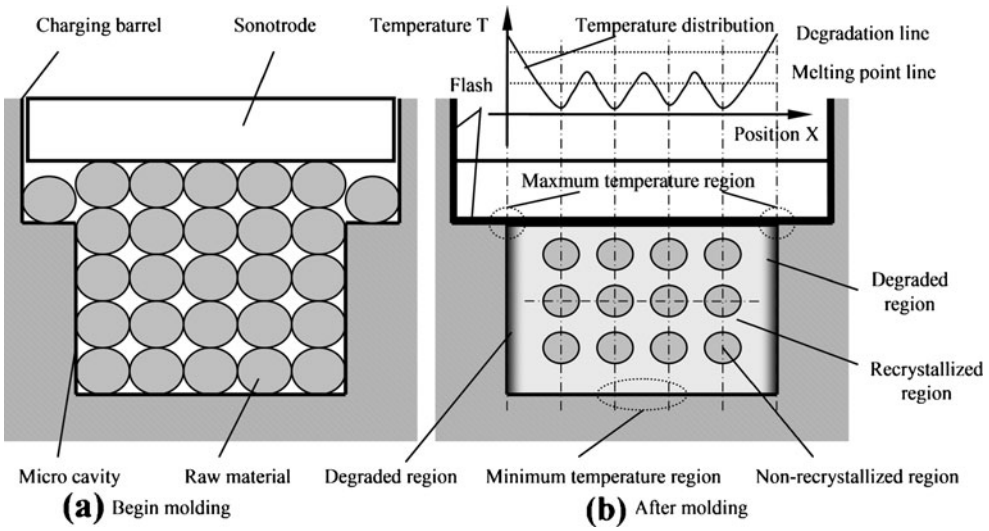

Fig. 5 Diagram of micro-UPM temperature distribution

(6 teeth, 3.6 mm pitch diameter, and 20° pressure angle) as shown in Fig. 6. The edge of the top gear with degraded black edges (Fig. 6a) has the highest temperature in the molding process. The results after flash removal and ultrasonic cleaning are shown in Fig. 6b. The processing parameters of the middle gear are relatively optimum (pressure of 0.1 MPa, ultrasonic time of 3.5 s, and holding time of 5 s). No black edges are observed in the microgear, the color is uniform, and the gear exhibits good plasticization.

To obtain qualified plastic parts, the temperature distribution should be controlled between the melting point line and degradation line, so that the nonrecrystallized region and the degraded region are removed and the pure recrystallization region is generated. The microplastic part volume is smaller, particularly in terms of height, the temperature difference between the top of lateral edges and the center of bottom surface is smaller, has a more uniform distribution of temperature, and the plastification quality is easier to control. Therefore, the method is particularly suitable for molding microplastic parts.

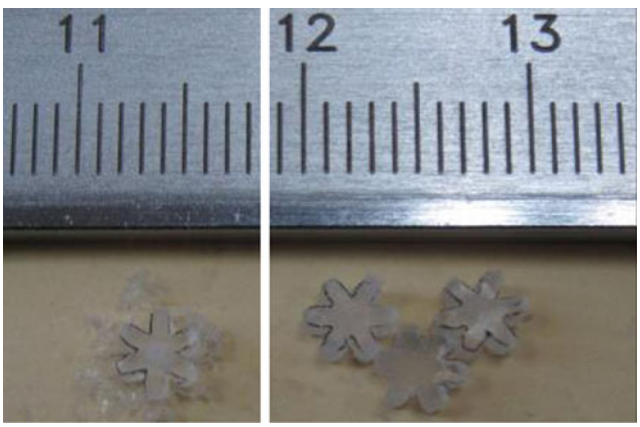

Fig. 6 Microplastic gears with degraded black flashes

3.2 Influence of process parameters on the flashes

The flash thickness is one of the important parameters for designing microplastic parts and controls the molding accuracy. To investigate the influence of pressure and ultrasonic time on flash thickness, an inlaid rectangular microcavity insert was designed for the experiment. The plasticizing cavity has dimensions of $2\times2\times1$ mm^3. Several experiments were performed at ultrasonic time ranging from 1.0 to 7.0 s under different pressure (0.1, 0.2, and 0.4 MPa, respectively). For every process parameter, more than five specimens were manufactured, and the flash thickness of these specimens were measured with a digital Vernier caliper, after which the average value was calculated (Fig. 7). The results indicate that under constant pressure, the flash gradually thins with increased ultrasonic time. Beyond the critical value, the flashes automatically separate from the microplastic substrate making them easier to remove. For example, 1.4 s is the critical value of ultrasonic time

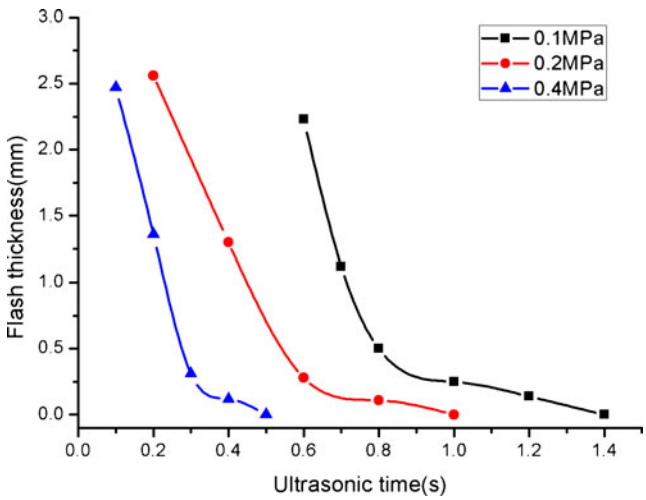

Fig. 7 Relationship among ultrasonic time, pressure and flash thickness (holding time of 5 s)

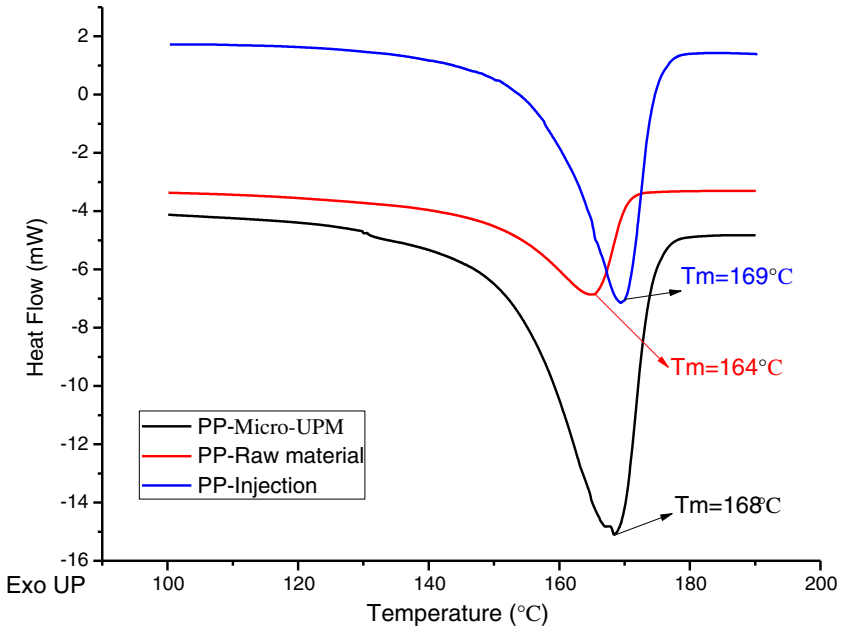

Fig. 8 DSC curves of samples

required for the flashes to automatically separate from the microplastic substrate under 0.1 MPa pressure (Fig. 7). The critical ultrasonic time is also considerably shortened with increased pressure because under higher pressure, the plasticizing capacity is greater and the flow rate of polymer melt is faster. Accordingly, shorter times are required to fill the microcavity and separate the flashes from the plastic substrate.

3.3 Thermal properties

The DSC curves of PP raw materials, micro-UPM, and injection samples are shown in Fig. 8. The following suitable micro-UPM processing parameters were used: pressure of 0.1 MPa, ultrasonic time of 1.4 s, and holding time of 5 s. The parameters of injection molding process were as follows: injection pressure of 80 MPa, injection temperature of 220 °C, mold temperature of 100 °C, and holding time of 6 s. The melting points and melting enthalpies of various samples can be measured through the DSC curve, and the crystallinity of various samples can be calculated based on Eq. (1). The results of DSC curve are listed in Table 1.

Based on the data in Table 1, the melting point of micro-UPM is around 168 °C, which is 4 °C higher than that of PP raw material and close to that of PP injection (169 °C). The crystallinities of various samples are similar. The crystallinity of plastic parts manufactured by micro-UPM is 50.2 %, which is 43 % higher than that of PP raw material and 19 % higher than that of PP injection. The results show that under the influence of ultrasonic vibration, the activity of molecular chain of PP powder is enhanced so that its crystal structure is considerably improved. Meanwhile, the noncrystallized region can be permeated into the crystal lattice and crystallized. Therefore, the melting point and the crystallinity are enhanced to some degree.

Table 1 DSC testing results of samples

Sample types	Melting point (°C)	Enthalpy (J/g)	Crystallinity (%)
PP raw material	164	73.5	35.1
PP micro-UPM	168	105.5	50.2
PP injection molding	169	88.0	42.1

3.4 Morphology and orientation analysis

In traditional injection molding, shear stress field and thermal field in the mold distribute in a gradient, and the molecular chain arrangement of parts injected by crystalline polymer has a hierarchical structure. Consequently, the parts exhibit a "skin–core" structure comprising a skin layer, a shear layer,

Fig. 9 Polarizing microscope photo of PP plastic parts

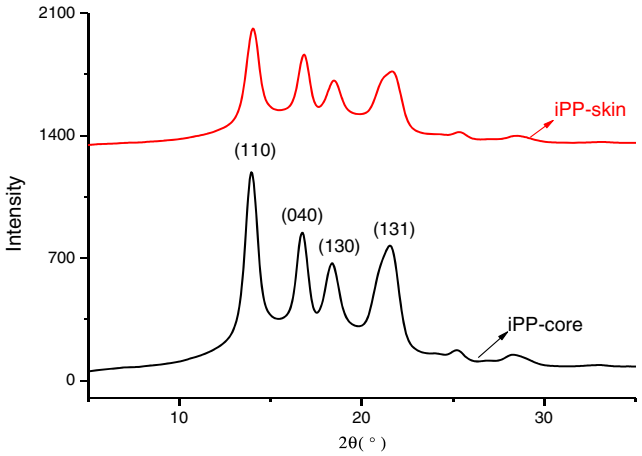

Fig. 10 X-ray diffraction pattern of the skin and core layer of micro-UPM samples

and a core layer [20, 21]. Polarized light microscopic observations reveal no obvious "skin–core" structure in the micro-UPM sample. Figure 9 shows the microscopic results, which reveal the presence of two distinct crystalline zones from skin to center: a skin layer and a core layer. No shear layer exists between these two layers because under the influence of ultrasonic conditions, the PP particles are directly melted in the mold cavity. Therefore, the PP particles do not pass through the fluid channel filling during molding. As a result, the shear effect of polymer melt is very small such that no shear layer exists in the internal organization. Therefore, a great difference in the morphological distribution is observed between microplastic parts obtained by micro-UPM and conventional injection molding.

Isotactic polypropylene is well known to exist in different crystalline forms: α, β, and γ crystal forms [17]. The α crystal form is the most common and the most heat-stabled crystal form of iPP. Figure 10 shows the diffraction profiles of different regions of the iPP microplastic part using an X-ray diffractometer. Each diffraction peak is assigned to a different crystallographic plane, which helps identify the crystalline forms in the material. Four evidently sharp diffraction peaks are observed in both skin and core layers. The intensities of the diffraction peaks are found at $2\theta = 14.1°$, 16.8°, 18.5°, and 22.6° corresponding to the (110), (040), (130), and (131) planes, respectively. The results indicate that only the α crystal form exists in the iPP microplastic part made by micro-UPM. Therefore, it can be inferred that there is no change in the crystal form of iPP microplastic parts made by micro-UPM, which greatly differs from that the α and β crystal form are both exist in the microstructure made by micro-injection molding [20].

Figure 11 shows the 2D single crystal X-ray diffraction (XRD) patterns of PP microplastic part molded by injection molding and micro-UPM, respectively. The crystal orientation of injection molding is very clear, whereas the crystal orientation of micro-UPM is not obvious, indicating that the two molding methods are obviously different.

3.5 Mechanical properties

Figures 12 and 13 show the changes in tensile strength, elongation at break of micro-UPM samples with ultrasonic time before and after annealing, respectively. The pressure of micro-UPM was 0.1 MPa, and the holding time was 5 s. The ultrasonic time was selected from the five time nodes as shown in Figs. 12 and 13. The temperature of annealing was 130 °C and the time was 60 min. Before and after annealing, with the extension of the ultrasonic time, the tensile strength and elongation at break of the samples have a trend of rise followed by drop. At a certain ultrasonic time, the tensile strength of various samples after annealing is higher than that of before annealing. Meanwhile, the elongation at break of various samples after annealing is lower than that of before annealing.

Analysis shows that when the ultrasonic time is 1.0 s, the energy provided by ultrasonic vibration is insufficient to plasticize the entire polymer microplastic parts in the microcavity. Along with the extending of ultrasonic time, the energy derived from the friction and compression among particles by ultrasonic vibration progressively increases, and the plasticizing effect of microplastic parts is improved. The optimum plasticizing effect of microplastic parts is reached when the ultrasonic time reaches 4.0 s. The microplastic parts molded at

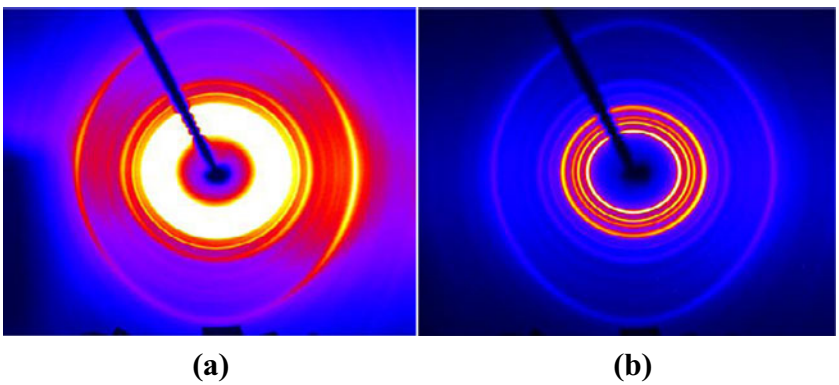

Fig. 11 2D single crystal X-ray diffractometer patterns of specimen from injection molding (**a**) and micro-UPM (**b**)

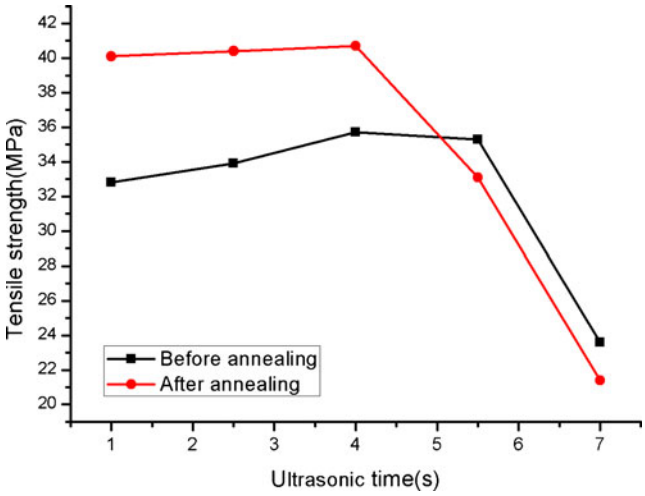

Fig. 12 Tensile strength versus ultrasonic time for before and after annealing

a certain ultrasonic time after annealing at the appropriate temperature can promote the thermal motion of polymer molecules, eliminate, or reduce the internal stress or structural defects to improve the mechanical properties of the products. Therefore, with increased ultrasonic time to a certain point, the plasticizing effect of microplastic parts gradually improves, and the tensile strength and breaking elongation rate increases. At the action of equal ultrasonic time, the mechanical properties of microplastic parts can be improved after annealing. However, when the ultrasonic time exceed more than 4.0 s, the polymer melt may be overheated and degraded, which seriously affects the mechanical properties of microplastic parts. Further heat treatment can only worsen rather than improve the mechanical properties of degraded products.

A set of optimum process parameters (pressure of 0.1 MPa, ultrasonic time of 4 s, holding time of 5 s) were selected according to the above analysis results. The tensile strength and elongation at break are compared with those of injection

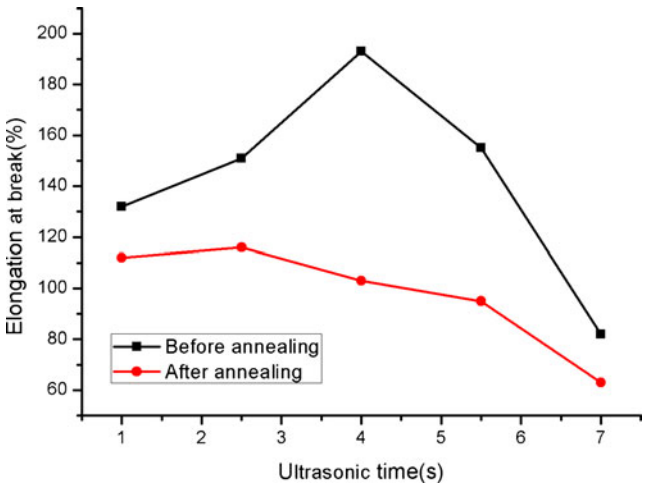

Fig. 13 Elongation at break versus ultrasonic time for before and after annealing

Table 2 Mechanical parameters of tensile samples

Sample types	Tensile strength (MPa)	Elongation at break (%)
Injection molding	33.3	650
Micro-UPM	35.6	193
Micro-UPM annealing	40.7	103

molding. The mechanical parameters are listed in Table 2. The tensile strength of micro-UPM is 35.6 MPa, which is 6.9 % higher than that of injection molding, and the elongation at break of micro-UPM is 193 %, which is only 29.7 % of the injection molding. After heat treatment of the micro-UPM samples, the tensile strength increases to 40.7 MPa, which is 14.3 % higher than that of unheated samples. The elongation at break of the heated samples is only 103 %, which is 46.6 % lower than that of unheated ones.

Analysis of experimental results shows that with the application of micro-UPM, the mechanical properties of samples are improved. Compared with conventional injection molding, the tensile strength of micro-UPM can be effectively improved, and the elongation at break is considerably decreased because the polymer melt is mainly affected by cavity shearing force during injection molding. However, the polymer melt in the microcavity is mainly influenced by three-dimensional compressive stress during micro-UPM molding so that micro-UPM samples have high compactness. Compared with injection molding, the stress state of micro-UPM is distinct and the tensile strength of micro-UPM samples is increased. After annealing at the appropriate temperature for micro-UPM samples, the residual stress of micro-UPM samples from the molding process is effectively improved, and the tensile strength of micro-UPM samples can be further improved.

4 Conclusions

A process called micro-UPM is proposed in the research. The molded samples confirm that the proposed method is a feasible and progressive technology. Based on the results and discussion, the following conclusions are drawn.

1. The polymer quickly melts and fills the entire microcavity, thereby greatly improving the problem of insufficient filling compared with conventional injection molding and avoiding the effects of weld marks on microplastic parts. In addition, the short molding cycle, low energy consumption, and low cost of the process makes it potentially useful in various studies and applications.
2. In the plastification molding process, the top outer edge of plastic parts is most likely to be degraded because it is subjected to the highest temperature, whereas the bottom center is most likely to be plasticized insufficiently

because it is subjected to the lowest temperature. Good quality plastic parts can be produced by controlling the molding temperature within the range of the melting point and degradation temperature of materials. Such method is suitable for molding microplastic parts.

3. Both the ultrasonic time and pressure significantly influence the flash thickness. Under the same pressure, the flashes gradually thin with increased ultrasonic time. After exceeding the critical value, the flashes can automatically separate from the substrate of microplastic parts. Increasing the pressure can significantly shorten the critical ultrasonic time for the separation of flashes.
4. The crystallinity and melting point of micro-UPM plastic parts increases by 2.4 and 43 % compared with those of PP powder, from 164 and 35.1 to 168 °C and 50.2 %, respectively. The melting point of micro-UPM plastic parts is close to that of the injection samples, but the crystallinity increases by 19 %, compared to that of the injection molding samples.
5. The crystal structure of micro-UPM sample does not show evident skin–core structure. XRD analysis shows that only the α crystal form exists in the iPP microplastic part. The crystal orientation of micro-UPM compared with that of injection molding is not obvious.
6. With increased ultrasonic time, the tensile strength and elongation at break of samples both have a trend of rise followed by drop before and after annealing. Within a certain ultrasonic time, the mechanical properties of various samples made by micro-UPM after annealing are improved. An ultrasonic time that is too long can cause the degradation of materials. The tensile strength of samples with optimum process parameters is 35.6 MPa (6.9 % higher than that of injection molding), and the elongation at break is 193 % (70.1 % lower than that of injection molding). After annealing under 130 °C for 60 min, the tensile strength of micro-UPM increases by 14.3 % and the elongation at break decreases by 46.6 %.

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