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Fabrication of microplastic parts with a hydrophobic surface by micro ultrasonic powder moulding

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phobicity.

ARTICLE INFO	A B S T R A C T
<i>Keywords:</i> Hydrophobic structure Micro ultrasonic powder Moulding UHMWPE	Microstructures with hydrophobic surfaces have been widely used in industrial fields such as self-cleaning and drag reduction. With the aim of fabricating microplastic parts with a hydrophobic surface, the paper processed the mould insert with micro-groove arrays from 304 stainless-steel plate through low-speed wire electrical discharge machining (LS-WEDM). And the micro-groove arrays were designed to U shape with bottom radius of 70 µm and depth of 110 µm. Ultra-high molecular weight polyethylene (UHMWPE) powders were used as raw material to fabricate microplastic parts with hydrophobic surface structures by micro ultrasonic powder

1. Introduction

Hydrophobic surfaces are receiving increasing attention because of their application in waterproofing, self-clean, anti-corrosion and drag reduction. Methods for fabricating hydrophobic surface structures mainly include electrolytic anodization [1], chemical deposition [2], the sol-gel method [3], etching [4] and the template method [5]. In the above techniques, the template method is a simple, effective and economical process, which can fabricate hydrophobic surface structures on a large scale. Because of low surface free energy and excellent moulding processability, polymer materials are widely used in processing of hydrophobic surface structures by the template method. Common moulding processes for polymer materials include hot embossing [6,7], injection moulding [8] and ultrasonic-assisted thermoplastic moulding [9].

Ultrasonic plasticization is a novel process for moulding of polymer materials. This method plasticizes polymer materials by ultrasonic vibration energy. In order to provide a reference for mould design and process development in ultrasonic microinjection moulding, *Jiang et al.* [10] investigated the influence of process parameters on the fluidity of the polymer melt. *Masato et al.* [11] studied the effects of ultrasound

melting on the mechanical and morphological properties of micropolypropylene parts and they found that the ultrasound injection moulding process could be an efficient alternative to the conventional process. Applying ultrasonic vibration in the polymer moulding process, the additional heat was not needed and the polymer materials were melted under the action of ultrasonic vibration. Therefore, the polymer materials were not processed under a high temperature environment for a long time, which reduces polymer degradation. Consequently, after ultrasonic vibration treatment, the microplastic parts have the characteristics of higher dispersion and higher level of crystallinity, whose mechanical properties can be improved [11]. In conclusion, ultrasonic plasticization method is suitable for the micro moulding of small materials. Liang et al. [12,13] used ultra-high molecular weight polyethylene (UHMWPE) powders as the moulding material and successfully prepared the micro-gear plastic parts by the micro-UPM process. The microstructure of microplastic parts prepared with different ultrasonic duration times was studied in detail by scanning electron microscopy (SEM).

moulding (micro-UPM). The parameters of the micro-UPM process were obtained by the single-factor test method. Under the ultrasonic energy of 1200 J, welding pressure of 100 kPa and pressure holding time of 8 s, microplastic parts with well surface morphology (Ra = 1.36μ m) and replication rate (97.76 %) were successfully fabricated. The surface contact angle of the microplastic part was 135.4°, which indicated well surface hydro-

The thermoplastic moulding process can fabricate not only polymer microplastic parts but also microstructures or micro-structured arrays with special functions on the surface of microplastic parts. By using

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Journal of Manufacturing Processes 56 (2020) 180–188

PLGA and polyvinylpyrrolidone (PVP) as raw materials, *Park et al.* [14] fabricated layered microneedles by spray deposition. The layered microneedles can effectively penetrate the skin for biphasic drug release. *Yin et al.* [15] prepared a two-dimensional nanofluidic device by hot pressing of a polymethyl methacrylate (PMMA) sheet. *Hu et al.* [16] prepared a microgroove lens array by hot embossing and applied the microgroove lens array to the LED light guide plate to improve the light transmission efficiency and uniformity.

Zaidi et al. [17] developed a dry-patching platform surface on the polyimide substrate. Cylindrical micropillar arrays were fabricated on the substrate surface by the polymer casting method to produce a hydrophobic surface with a self-cleaning property. Rasilainen et al. [18] prepared a hydrophobic micro-structured surface on polypropylene by injection moulding. HuyNguyen et al. [19] demonstrated the fabrication of a polyurethane-acrylate (PUA)-based hydrophobic surface with a water contact angle of 146°. First, the PUA liquid was drop-dispensed onto the mould and then the microarray structure on the mould was replicated on the PUA surface. Finally, the PUA surface was modified with a low-surface-energy siloxane. Yoon et al. [20] obtained a microstructured polydimethylsiloxane (PDMS) film with a contact angle in the range of 157° to 161° by the etching method. Minarik et al. [21] created nanostructures on the surface of polystyrene by tetrafluoromethane (CF4) plasma modification. The contact angle of the modified polystyrene surface exceeded 155°. Brown et al. [22] prepared an ordered honeycomb surface on a polybutadiene film by the solvent casting method. The film was made superhydrophobic by the subsequent CF4 plasma modification. Zhi et al. [23] embedded surfacefunctionalized silica nanoparticles on an epoxy resin substrate to make the substrate surface superhydrophobic.

In this study, the micro ultrasonic powder moulding (micro-UPM) process was adopted to fabricate microplastic parts with a hydrophobic surface. The mould insert with micro-groove arrays was fabricated from 304 stainless-steel plate by the low-speed wire electrical discharge machining (LS-WEDM). The micro-groove arrays were designed to U shape with bottom radius of 70 μ m and depth of 110 μ m. UHMWPE powders were selected as the raw material. Under effect of suitable process parameters in micro-UPM, the raw material powders were melted and rapidly filled into the mould cavity to fabricate microplastic parts. After the relevant equipment detection, it was found that the microplastic parts had well moulding quality, high replication rate and well hydrophobicity.

2. Experimental

2.1. Materials

The raw material is an ultra-high molecular weight polyethylene (UHMWPE) powder (Z1700, SAMSUNG) with an average relative molecular weight of 3.5 \times 10⁶. The average particle size is approximately 150 μm .

2.2. Equipments

The experimental equipments used in this paper are shown in Fig. 1. The mould insert (15 mm \times 15 mm \times 2 mm) with micro-groove arrays was fabricated from 304 stainless-steel plate by LS-WEDM (AP250LS, Sodick, Japan). The micro-groove arrays were designed to U shape with bottom radius of 70 µm and depth of 110 µm. From measurement results, the bottom radius of the obtained micro-groove was from 68.5 µm to 69.8 µm and the depth of micro-groove was from 108.2 µm to 119.5 µm. The mould consisting of an upper plate, a lower plate and a mould insert was fixed on the ultrasonic welder (2000XCT, Branson, USA) to fabricate microplastic parts. A through hole with diameter of 10 mm was processed in the upper plate, which was used as moulding cavity. A rectangular groove (15 mm \times 15 mm \times 2 mm) was processed in the lower plate and it was used as mould insert cavity. The surface morphology, normal cross-sectional area and surface roughness were characterized by a laser confocal microscope (VK-X250 K, Keyence, Japan). The static contact angle of droplets on the microplastic part surface was measured with drop shape analyse (DSA100S, Krüss, Germany) under the temperature of 26 $^{\circ}$ C.

2.3. Process flow

The micro-UPM process for preparing surface hydrophobic microplastic parts is shown in Fig. 2 and its process flow is described as follows.

(1) The 304 stainless steel was placed on the LS-WEDM (Fig. 2a) and then it was processed to obtain mould insert with micro-groove arrays (Fig. 2b). (2) The mould insert obtained from step 1 was installed into the mould insert cavity and then it was fixed in the ultrasonic welder. (3) The UHMWPE powders were filled into the mould cavity (Fig. 2c and Fig. 2d) and compacted by moving the sonotrode up and down (Fig. 2e). (4) After setting the ultrasonic process parameters, the ultrasound was applied to the UHMWPE powders (Fig. 2f). Under the actions of ultrasonic energy and welding pressure, the UHMWPE powders were rapidly melted and filled into the mould cavity. (5) After a certain pressure holding time (Fig. 2g), the replication of the microgroove arrays was completed and a microplastic part with surface hydrophobicity was thereby obtained (Fig. 2h).

During micro-UPM, ultrasonic vibration was applied to the UHMWPE powders and the ultrasonic vibration was directly related to ultrasonic energy. Under the effect of the ultrasonic vibration, friction between the UHMWPE powders could take place and thus the heat can be generated for fabricating the microplastic parts. Therefore, the ultrasonic energy has a same effect to mould temperature and different ultrasonic energies represent different mould temperatures. Under the effect of the ultrasonic vibration, the UHMWPE powders were melted. Then, under the effect of welding pressure, the melt was quickly filled into the mould cavity. For ensuring the moulding quality of microplastic parts, the welding pressure should be maintained for a certain time and this process was called as pressure holding. The certain time was called as pressure holding time.

3. Influence of micro-UPM on moulding quality of microplastic parts

In the micro-UPM process, the process parameters of ultrasonic welding have important influences on the quality of the microplastic parts. The process parameters of the ultrasonic welding included ultrasonic energy, welding pressure, and pressure holding time. In this paper, the single factor experiment method was used to determine the appropriate process parameters. For investigating the stability of process parameters, 5 microplastic parts were fabricated under each group of process parameters. The moulding quality of microplastic parts was mainly evaluated by the replication rate and the surface morphology.

3.1. Influence of ultrasonic energy on moulding quality of microplastic parts

In the paper, the micro-UPM used an ultrasonic welder to fabricated microplastic parts. During the micro-UPM, the ultrasonic welder was set in the energy control mode and ultrasonic energy can be inputted through the instrument control panel. The ultrasonic energies used in experiments were 800 J, 1200 J, 1600 J and 2000 J respectively. The pressure holding time and welding pressure were kept at 8 s and 100 kPa respectively. The experimental results are shown in Fig. 3.

When the ultrasonic energy was 800 J, the ultrasonic energy was insufficient and the UHMWPE powders existed in the form of lumpy particles that adhered to one another. Under this circumstance, there were a lot of marks on the surface of microplastic parts (Fig. 3a). With the ultrasonic energy increased, the marks on the surface of microplastics gradually decreased (Fig. 3b). When the ultrasonic energy was

Journal of Manufacturing Processes 56 (2020) 180-188



Fig. 1. The experimental facilities used in this paper.

1600 J, the marks on the surface of microplastic part were almost invisible (Fig. 3c). However, if the ultrasonic energy continued to increase, the microplastic part can be broken and the crack would appear on the surface of microplastic (Fig. 3d).

During micro-UPM, ultrasonic energy was applied to the polymer powders and the friction between the polymer powders could take place, which can generate the heat for fabricating the microplastic part. Therefore, with the ultrasonic energy increased, more and more UHMWPE powders were melted until all the UHMWPE powders were melted completely, which resulted in improvement of moulding quality of the microplastic part. However, Excessive ultrasonic energy can damage the surface of microplastic part.

3.2. Influence of welding pressure on moulding quality of microplastic parts

In micro-UPM, the ultrasonic welder was used to fabricate microplastic parts. Before the experiment, the different welding pressures can be inputted through the instrument control panel. The welding pressures used in experiments were 70 kPa, 100 kPa, 130 kPa and 160 kPa respectively. The pressure holding time and ultrasonic energy were kept at 8 s and 1200 J respectively. The experimental results are shown in Fig. 4.

When the welding pressure was low, part of the ultrasonic energy would be lost on the sonotrode. Under this circumstance, friction

between UHMWPE powders can not take place adequately and thus the heat required for UHMWPE powders fusion was insufficient. Consequently, the UHMWPE powders were melted locally and stick marks appeared on the surface of the micro plastic parts (Fig. 4a). When the welding pressure was 100 kPa, the UHMWPE powders were fully melted and the surface morphology of the micro plastic parts was well (Fig. 4b). With the welding pressure increased to 130 kPa, the welding pressure was higher, which can restrict the relative movement between the UHMWPE powder. In this case, friction between UHMWPE powders cannot effectively generated and thus the heat required for UHMWPE powders fusion was insufficient. As a result, the UHMWPE powders were melted insufficiently and stick marks appeared again on the surface of the micro plastic parts (Fig. 4c). When the welding pressure was 160 kPa, the ultrasonic welding process cannot proceed smoothly because of the excessive welding pressure. Under the impact of the excessive welding pressure, the surface of the microplastic part was easily damaged (Fig. 4d).

3.3. Influence of pressure holding time on moulding quality of microplastic parts

During micro-UPM, the welding pressure was firstly applied to the UHMWPE powders and then the ultrasound was applied to the UHMWPE powders. Under the actions of ultrasonic energy and welding



Fig. 2. Schematic representation of the process flow. (a) Processing of mould insert; (b) Mould insert; (c) Ultrasonic welder; (d) Cavity filling; (e) Compacting UHMWPE powders; (f) Applying ultrasonic vibration; (g) Pressure holding; (h) Microplastic part with surface hydrophobicity.

pressure, the UHMWPE powders were rapidly melted and filled into the mould cavity. After that, the ultrasound stopped and the welding pressure must be hold for a certain time to ensure the moulding quality of the microplastic part. This time was called as pressure holding time. The pressure holding times used in experiments were 2 s, 4 s, 8 s and 16 s respectively. The ultrasonic energy and welding pressure were set as 1200 J and 100 kPa respectively. The results are shown in Fig. 5.

When the pressure holding time was 2 s, the microplastic part was not fully formed. In this case, the melt did not cool completely and thus part of the melt was pulled out of the mould cavity with the reset of the sonotrode, which caused damaged area to appear on the surface of the microplastic part (Fig. 5a). With the holding time increased to 4 s, the melt did not cool completely and there were some depressions on the surface of the microplastic part (Fig. 5b). With the pressure holding time increased to 8 s and 16 s, the melt cooled completely and surface morphology of the microplastic part was well (Fig. 5c and Fig. 5d). However, When the pressure holding time was too longer, deformation of the microplastic part would occur.



Fig. 3. The micro-UPM results of microplastic parts under different energies: (a) 800 J; (b) 1200 J; (c) 1600 J; (d) 2000 J.



Fig. 4. The micro-UPM results of microplastic parts under different welding pressures: (a) 70 kPa; (b) 100 kPa; (c) 130 kPa; (d) cross-section profile of Fig. 4c; (e) 160 kPa.



Fig. 5. The micro-UPM results of microplastic parts under different pressure holding time: (a) 2 s; (b) 4 s; (c) 8 s; (d) 16 s.

4. Results

4.1. Replication rate

To evaluate the quality of microplastic parts, six samples from section 3 with better surface morphology were selected for the

comparison of cross-sectional replication rate. The selected samples were shown in Fig. 3b, Fig. 3c, Fig. 4b, Fig. 4c, Fig. 5c and Fig. 5d. The paper named the above samples as 1#, 2#, 3#, 4#, 5# and 6# respectively. In the paper, the cross-sectional replication rate was defined as the ratio of S_1/S_2 , where S_1 denoted the cross-sectional area of the microplastic part and S_2 denoted the corresponding cross-sectional area

Sample	Cross-sectional area of mic	Mean replication		
Sample	measurement diagram $S_1 (mm^2)$		rate	
1# (Show in Figure 3b)	100.000 0.000 0.000 200.000 µm	15153.1	97.51%	
2# (Show in Figure 3c)	100.000	14853.4.1	95.58%	
3# (Show in Figure 4b)	100.000 0.000 0.000 200.000 µm	15192.9	97.76%	
4# (Show in Figure 4c)	100.000 0.000 0.000 <u>200.000 µm</u>	14750.3	94.92%	
5# (Show in Figure 5c)	100.000 - 100 - 	15170.3	97.62%	
6# (Show in Figure 5d)	100.000 0.000 0.000 200.000 µm	14745.2	94.88%	

Fig. 6. The cross-sectional replication rates of the 6 microplastic parts.

of the mould insert. 5 microplastic parts were fabricated under each group of process parameters and the unique mould insert was used for each group of process parameters. 5 different positions in every

microplastic part and mould insert were randomly selected to measure the cross-sectional areas by laser confocal microscope and its software (VK MultiFile Analyzer). From the measurement results, arithmetic



Fig. 7. The measurement baselines of microplastic part and mould insert.

mean of the cross-sectional area can be obtained, which can improve the calculation accuracy of the cross-sectional replication rate as much as possible. The measurement baselines of microplastic part and mould insert are shown in Figs. 6 and 7 and the cross-sectional replication rates of the microplastic parts are shown in Fig. 6.

According to the experimental results shown in Fig. 3, Fig. 4 and Fig. 5, more and more UHMWPE powders were melted and the interparticle voids were gradually reduced under the appropriate process parameters. Therefore, the stick marks on the surface of microplastic

Table 1			
The roughness of mould	insert ar	nd microplastic	parts

Sample	Ra (µm)	
	convex	concave
Mould insert	2.81	2.62
Microplastic part 1#	1.39	1.64
Microplastic part 3#	1.42	1.57
Microplastic part 5#	1.36	1.63

part gradually disappeared and the moulding quality of microplastic parts was improved. From the measurement results shown in Fig. 6, the cross-sectional replication rate of microplastic part 1# (Fig. 3b) is higher than that of 2# (Fig. 3c) in the ultrasonic energy group. The cross-sectional replication rate of microplastic part 3# (Fig. 4b) is higher than that of 4# (Fig. 4c) in the welding pressure group. The cross-sectional replication rate of microplastic part 5# (Fig. 5c) is higher than that of 6# (Fig. 5d) in the pressure holding time group. Therefore, it can be concluded that UHMWPE microplastic parts with well moulding quality can be produced via micro-UPM by using the following parameters: ultrasonic energy of 1200 J, welding pressure of

Sample	Surface topography		Roughness profile
Mould	Measurement position	concave	14.772
insert	<u>200 шт</u>	convex	13.106 0.000 -13.106 -13.00
1#	Measurement position	convex	9.29 0.000 9.29 9.29 0.0000 0.0000 0.0000 0.000 0.000 0.000 0.000 00
(Show in Figure 3b)	200 μm	concave	8.991 0.000 -8.991 0.000 200,000 400,000 600,000 600,000 600,000 600,000 1099,398 1097 100
3#	Measurement position	convex	9.112 0.000 -9.112 0.000 200.000 400.000 600.000 800.000 1060.090
(Show in Figure 4b)	<u>200 µт</u>	concave	7.015 0.000 -7.015 0.000 200.000 400.0000 400.000 400.0000 400.0000 400.0000 400.0000 400.0000 400.
5#	Measurement position	convex	9, 229
(Show in Figure 5c)	<u>200 µm</u>	concave	8.991 0.000 0.000 0.000 200.000 400.0000 400.000 400.0000 400.0000 400.0000 400.0000 400.0000 400.0

Fig. 8. The roughness of the mould insert and microplastic parts.



Fig. 9. The contact angle of the mould insert and microplastic parts: (a) Mould insert; (b) Microplastic part 1#; (c) Microplastic part 3#; (d) Microplastic part 5#.

100 kPa and holding pressure of 8 s.

4.2. Roughness

From above experimental results, it can be known that microplastic part 1# (Fig. 3b), microplastic part 3# (Fig. 4b), and microplastic part 5# (Fig. 5c) have well surface morphology and replication rate. The line roughness Ra of the above samples was measured by the laser confocal microscope. The above Ra values were measured at convex surface and concave surface of the microgroove of the mould insert and microplastic parts (Fig. 8). Each Ra value was measured 5 times in the different convex surfaces and concave surfaces and then the measurement data was averaged to obtain the final Ra value. During the measurement, the shape error was removed. The measurement results are shown in Table 1 and Fig. 8. The mean Ra of the concave region in the mould insert is 2.62 µm (Table 1). The mean Ra of the corresponding convex region in the microplastic parts is all less than 1.42 µm (Table 1), which is 45.8 % lower than Ra of the mould insert. The Ra of the convex region in the mould insert is 2.81 µm (Table 1). The Ra of each corresponding concave region in the microplastic parts is less than 1.64 μ m (Table 1), which is 41.6 % lower than the Ra of the mould insert. According to the experimental results, the roughness of the microplastic parts is smaller than that of the mould insert.

The reasons for the above results are as follows. When the mould insert was machined by the LS-WEDM, a pit-protrusion-porosity-flake composite rough microstructure was constructed on the surface of the mould insert. The composite rough microstructure was not filled completely by the molten UHMWPE under the comprehensive effects of ultrasonic energy and welding pressure. Therefore, the microplastic part fabricated by the micro-UPM process had a smaller Ra than that of mould insert.

4.3. Hydrophobicity

The contact angle of water on the surface of solid material is an important parameter to measure the hydrophobicity on the surface of material. Generally, a material is hydrophilic when the contact angle is less than 90° and a material is hydrophobic when the contact angle is more than 90°. With the aim of fabricating microplastic parts with a hydrophobic surface, the research fabricated microplastic parts by micro-UPM. In order to investigate the hydrophobicity on the surface of microplastic parts, the parallel contact angle was measured. 5 different positions in every microplastic part and mould insert were randomly selected to measure the static contact angle of droplets. Fig. 9a shows that the mean contact angle of the mould insert is 129.1°. The mean contact angles of microplastic part 1# (Fig. 9b), microplastic part 3# (Fig. 9c) and microplastic part 5# (Fig. 9d) are 135.4°, 132.9°, and 130.2° respectively. The above results show that the mean contact angles of the microplastic parts are larger than that of the mould insert and the surface of the microplastic parts has well hydrophobicity. The above experimental results indicate that micro-UPM can be used to prepare microplastic parts with hydrophobic surfaces.

The surface hydrophobicity is mainly attributed to the rough surface with special microstructures and the low surface free energy. The surface energy of water is large. The droplet cannot be spread on low surface energy materials and remains basically in the form of a sphere, which shows hydrophobicity [17]. The micro-grooves are processed by a low-speed wire electrical discharge machine, the secondary nanostructured is also processed on the surface of the micro-grooves. This co-existence of micro-nano structures may increase the specific surface area of the sample and provide more air trapping, which makes the material hydrophobic [24]. The microplastic parts have almost the same microstructures as the metal mould insert. However, polymer materials have lower surface free energy than metal materials. The droplet did not spread on the surface of the microplastic parts and remained basically in the form of a sphere. Therefore, the contact angle of the droplet on the microplastic part is larger than that on the mould insert.

5. Conclusions

In this paper, A mould insert with micro-groove arrays was fabricated by LS-WEDM. According to the mould insert and using UHMWPE powders as raw material, microplastic parts with surface hydrophobicity were successfully prepared by the micro-UPM process. The main conclusions of this study are as follows:

- (1) Under the ultrasonic energy of 1200 J, welding pressure of 100 kPa and pressure holding time of 8 s, microplastic parts with surface roughness Ra of 1.36 μ m and the replication rate of 97.76 % were prepared by micro-UPM process.
- (2) The maximum and minimum contact angles of the microplastic parts prepared by micro-UPM were 135.4° and 130.2° respectively. The droplet did not spread on the surface of the microplastic parts and remained basically in the form of a sphere, which indicated well surface hydrophobicity. Micro-UPM provided a simple and quick method for fabricating microplastic parts with surface hydrophobicity.
- (3) The single factor experiment method was used to determine the appropriate process parameters and the iteration effect between process parameters was considered in the paper. In the future research, the authors will discuss the iteration effect and use it to optimize the process parameters of the micro-UPM. Moreover, microchannel inserts with different cross-section shapes and simulating their moulding process with FLUENT software will be studied in detail.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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