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Injection molding of micro pillars on vertical side walls using polyether-ether-ketone (PEEK)

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Abstract

This paper investigates the replication of microstructures on a vertical wall by PEEK injection molding. A 4-cavity insert was used in the injection molding. Pre-fabricated nickel plates with $\varnothing 4 \mu\text{m}$ micro holes on the surface were glued on vertical walls in the cavities. 3 cavities were coated by CrN, TiN and TiB₂ respectively, the remaining one was not coated as a reference. The effect of coating was compared via the morphology of the micropillars on the polymer parts. 4000 injection molding cycles were repeated. The roughness of the coated surface was measured. The reasons for the demolding result were discussed.

Keywords: Micro pillars, PEEK injection molding, vertical side, 3D

1. Introduction

Functionalities realized by micro-structured surfaces are applied in many areas, for example in the field of cell biology [1] [2] optical elements [3], etc. The micro structured surface is of special interest to the authors due to the fact that it can promote cell proliferation and enhance the bonding between the tissues and the surface, when the micro-features are arranged in a certain way [4]. In order to study the feasibility of micro-structuring on the surface of a complex geometry, in this paper, replication of micro pillars on a vertical side wall is investigated.

There are multiple reports on fabricating microstructures on a surface as well as replicating them by polymer, glass or other materials. Most of the work focuses on flat surfaces or surfaces with a limited curvature. Processes for structuring a surface with a continuous curvature were also reported, for instance S. Bruening et al. implemented a cylinder processing system [5]. However, structuring and replication on the vertical side is rarely reported, although most of the real-life devices, that need micro structuring on the surface, have a three-dimensional geometry. There are huge demands for investigations in this area, even though there are challenges for both establishing the micro structures on the surface of a

3D mold and the replication.

Replication on a vertical side wall was mentioned in [6], where the surface structures are in the sub-micro range. The micro pillars reported in this paper are approximately $2 \mu\text{m}$ high. The previous work of the authors replicated the same micro pillars on a 3D cavity by liquid silicon rubber using injection molding [7]. The good elasticity of the liquid silicone rubber (LSR) reduced pillars breaking during the demolding process.

In this paper, injection molding was operated on a vertical side wall (90 degree) with micro holes. The injected material polyether ether ketone (PEEK) is well-known for its high stiffness. The difficulty for demolding was increased. The micro-structured surface was obtained from a prefabricated Ni plate, which was cut into small pieces and glued on the insert to form both horizontal and vertical sides. Injection molding of PEEK using the same type of Ni plates was tested in the authors previous research on flat specimens [8]. It was demonstrated that for the studied surface pattern, high impulse magnetron sputting - physical vapor deposition (HIPIMS-PVD) CrN coating improved the demolding. In this paper two further types of coatings, TiN and TiB₂, were tested.

The purpose of this research is to demonstrate the

possibility of micro pillar's replication on a complex mold with 3D shape. Furthermore, the wear of the inserts was investigated over 4000 injection molding cycles for all insert types.

2. Experiments

2.1 Tooling

With the purpose of creating micro pillars on the surface, a prefabricated nickel plate with micro holes on the surface was cut by EDM and then glued on the surface of the mold insert. The Ni plate was fabricated by Hoowaki® using a lithographical process [9] [10]. Figure 1 shows the surface structure on the Ni plate. The micro holes were 4 μm in diameter and 2 μm deep, with a 2 μm edge-to-edge distance and placed in square lattice. The holes have a 1 degree draft angle which facilitates demolding. This pattern was approved by fibroblast cell proliferation test. Figure 2 shows how they are arranged to form vertical and horizontal surfaces for the polymer parts.

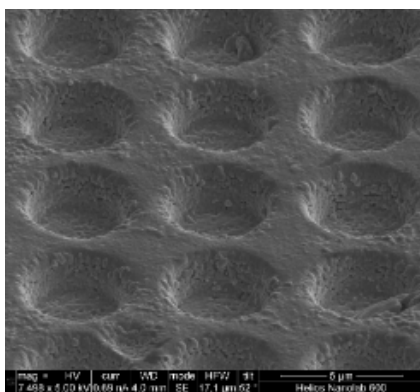


Figure 1 The surface structure on the nickel plate. The holes are Ø4 μm and 2 μm deep.

A four-cavity mold was used. A steel insert was mounted on the counter plate; four cavities were made on the insert, as illustrated by Figure 2; Inside the insert cavities, Ni pieces were glued by epoxy from EPO-TEK® 353ND; Coatings were applied on the Ni pieces before gluing. Figure 5 shows the mold opening direction, which is vertical to the highlighted surface.

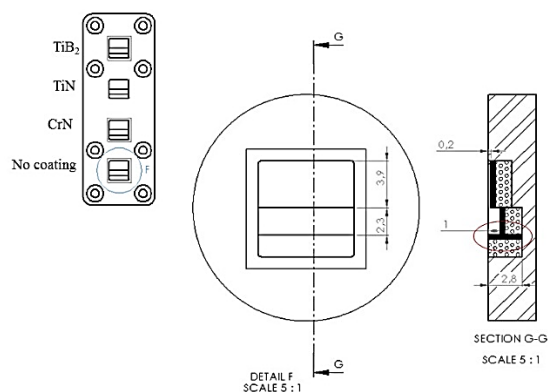


Figure 2 illustration for the insert cavities. In each cavity, 3 pieces of structured Ni plates are glued and they form horizontal and vertical sides. The red circle highlights the Ni plate which leads to a vertical side on the polymer part. The bold lines show the surface with micro holes.

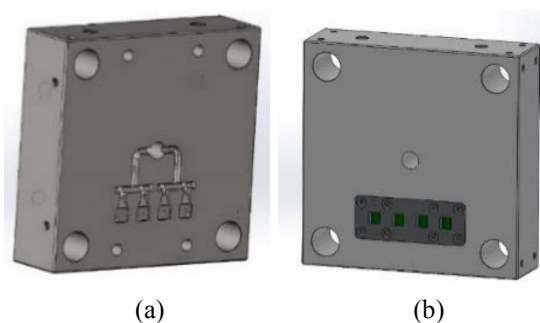


Figure 3 the mold plates with 4 cavities and the insert on the fixed plate also has 4 cavities. (a) the movable plate (b) the fixed plate.

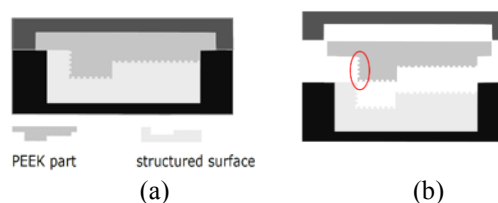


Figure 4 (a) The mold is filled by PEEK (b) the mold is open. The red circle highlights the investigated side, which is vertical to the mold open direction.

2.2 Injection molding process

4000 injection molding cycles were operated by an Arburg (370 A 600 – 70) injection molding machine with a reciprocating screw with a diameter of 18 mm and a clamping force of 300 KN. PEEK Vestakeep® 1000G from Evonik was used for the experiments. It is an unreinforced PEEK grade with

low viscosity. A mold temperature of 190°C was set. The injection speed used was 175 mm/s and the melt temperature was 390°C. An injection pressure of 900 bars was reached. A total cycle time of 29s was obtained including packing, cooling and demolding phases. After 4000 cycles of PEEK injection molding, half of the epoxy lost function (after a two week pause, when re-heated up, they lost function) i.e. the Ni plates in two cavities peeled off.

2.3 Coating

The films were deposited by non-reactive- and reactive HiPIMS, and pulsed DC magnetron sputtering, in a commercial batch-coating system (CC800/9, CemeCon AG) on microstructured Ni-inserts and 1.5 × 1.5 cm² Si (001). The substrates were mounted on a substrate table carrying out a two-fold planetary rotation[11]. The target-substrate distance was approximately 8 cm.

Table 1: Major parameters for coatings

Coating	Tech.	T °C	P mPa	Thickness nm
CrN	Reactive HiPIMS	200	330	200
TiB ₂	HiPIMS	550	430	100
TiN	DC	500	520	200

Prior to depositions, the chamber was evacuated to a base pressure below 1 mPa and the samples were pre-heated to prevent outgassing during deposition. Table 1 lists the deposition conditions, including the specific technique used for each type of coating, the approximate deposition temperature and pressure, and the expected approximate thickness of the coating. Prior to coating deposition, plasma etching is used to clean the surface; it is used to improve the bonding of the deposited coating on the substrate. Different etch parameters were used for the different coatings, and may influence the substrate roughness.

3. Result and analysis

3.1 The surface roughness before and after coating

The roughness of the Ni plates was measured before any injection molding. Olympus Lext OLS4100 3D Laser Measuring Microscope (100x) was used to measure on the Ni pieces with and without coatings. SPIP was used to analyze the roughness of the bottom of the holes and the top of the surfaces. Obviously the coatings reduced the roughness, as showed by Table 2.

Table 2 The Sa values of the Ni plates

Sa (nm)	Bottom of holes	On top surfaces
no coating	155 ± 13	84 ± 27
CrN	54 ± 10	51 ± 19
TiN	49 ± 9	56 ± 16
TiB ₂	141 ± 10	61 ± 15

3.2 Wear of coating

The wear of the coatings was investigated by possible decrease in thickness. The thickness was examined by a coating thickness analyzer Oxford Xstrata-980. The measurement resolution of the instrument is 0.1 μm. The thickness of the coating is believed to be unchanged after 4000 shots. No wear could be identified using this method. This result agrees with our previous conclusion where CrN coating was tested for 6000 cycles by PEEK injection molding under the same conditions.

3.3 Replication of the pillars on the vertical wall of the polymer parts

During injection molding, 5 samples were taken out after cycle 600, 1300 and 2500 respectively. The vertical walls were studied by scanning electron microscope (SEM). The topography of the pillars on a random position of the vertical wall is displayed by Table 3. Nevertheless, they present the major topography. Pillars from far from the gate and close to the gate do not show any significant difference concerning the shape.

In cycle 600, the pillars are mostly demolded successfully, i.e. no pillars breaking or other defects. Except, most pillars from the TiN coated cavity missed part of the top. In cycle 1300 and 2500, larger area from the pillars from TiN cavity were lost. As concluded in our previous research [8], when trapped polymer starts to occur in some holes, more tends to happen in those holes in the future cycles.

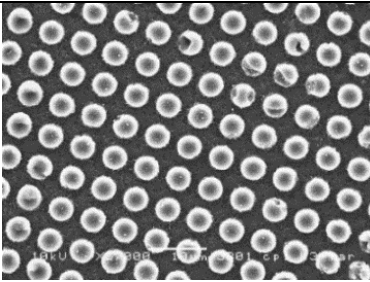
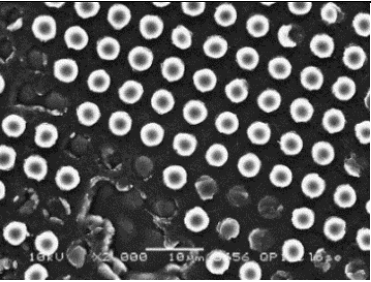
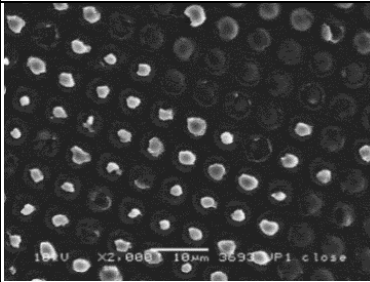
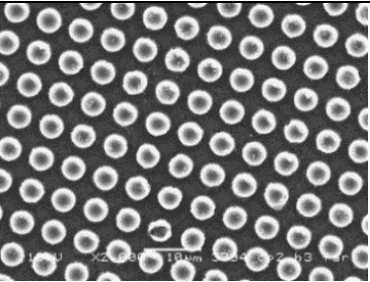
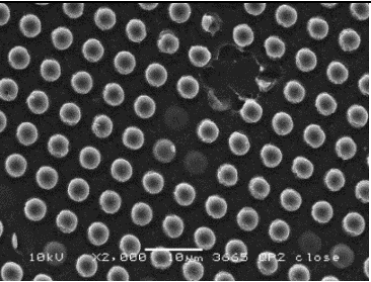
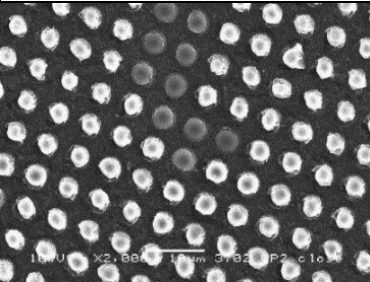
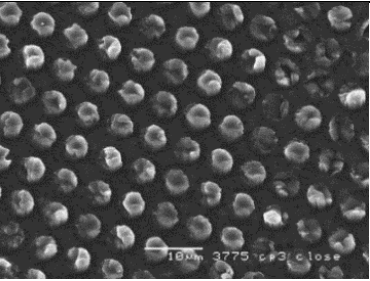
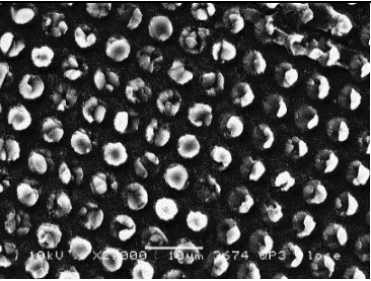
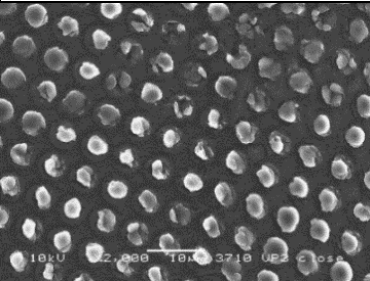
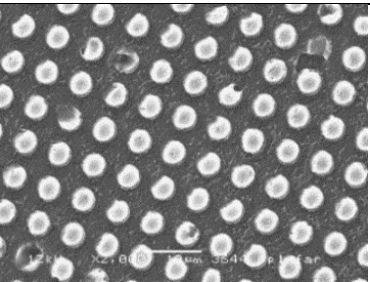
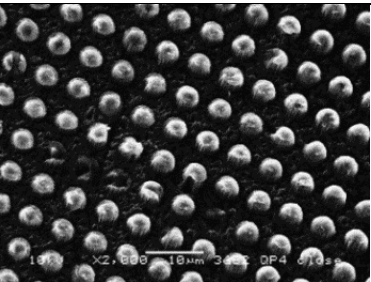
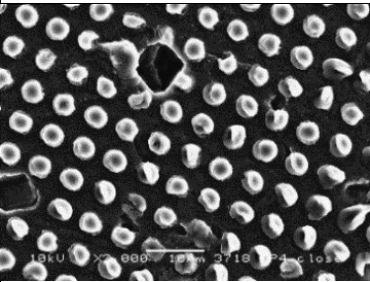
Pillars broke occasionally in the cavity without coating in cycle 1300; most of the existing pillars still have a good shape.

Compared with the horizontal surface, pillar breaking happens in earlier cycles. There are also much more failures in cycle 2500, compared with similar cycles for the horizontal samples.

For cycle 2500, more defective pillars are observed, especially for samples without coating. Broken pillars, partly or completely, are much less when CrN or TiB₂ coating is applied on the inserts. No major difference can be identified on the pillars when using these two coatings.

There are many possible reasons that can lead to demolding failures for the pillars. However the roughness is excluded because after coating the roughness (Sa) in the holes and on the top surface is lower than before coating. According to Tosello et. al.

Table 3 Topography of the vertical side on the PEEK parts

	Cycle 600	Cycle 1300	Cycle 2500
No coating			
CrN			
TiN			
TiB2			

[12], TiN coating has a slightly higher coefficient of friction (COF) compared to Ni surface. However the Hoowaki inserts has a layer of silver on the top, and the COF of silver is much higher than Nickel or the TiN coating, so it does not explain the cavity without coating leads to better demolding than with TiN coating.

The depth of the holes when using TiN coating is 100nm larger than using the other coatings or without any coating. Though it won't change the aspect ratio significantly, it probably can partly explain the unsuccessful demolding.

Another possible reason for the unsuccessful filling in the cavity is unbalanced runner system for

the four cavities. Due to the micro size of the surface features, the surface area is significantly increased. If the flow to the last two cavities with TiN and TiB2 coatings are slightly slower, it is enough to freeze the polymer flow and lead to a failure in filling the micro holes. A short shot test will release the flow rate in the runner system. However this work will be leave for future due to the lack of time.

The height of the pillars were measured by a OLYMPUS LEXT 3D measuring laser microscope. The height of the pillars remain unchanged for each cavity during the entired 4000 shots considering variation. This result agrees with the previous study on CrN coated cavity for flat samples.

4. Conclusions

More than 4000 cycles of micro pillars injection molding were operated using cavities with both horizontal and vertical sides. CrN, TiN and TiB₂ coatings deposited by HIPIMS PVD were applied in the cavities and compared to a Ni insert without any coating. The roughness of the inserts were reduced after all the coatings. For the first 1000 cycles, pillars demolded from the vertical side with only few failures except the cavities with TiN coating. After 2500 cycles, CrN and TiB₂ coating improve demolding when the form of the pillars is concerned. Most of the pillars demolded successfully up to 3000 cycles when using CrN and TiB₂ coatings.

5. Acknowledgement

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