



A novel texturing of micro injection moulding tools by applying an amorphous hydrogenated carbon coating

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ABSTRACT

This paper investigates the effect that a novel texturing of replication tools by applying amorphous hydrogenated carbon (aC:H) coating has on the processing conditions in micro injection moulding. Texturing usually increases the surface area of tools leading to higher demoulding forces acting on the component during the ejection stage of the process. As a consequence of this the ejection forces can cause stress marks, deformation, fracture and stretching of the polymer micro features. Therefore, this research studies the effect that a textured aC:H coating with nano scale structures has on the resulting demoulding forces in comparison to an untreated tooling surface. The obtained results demonstrate the beneficial effect of the aC:H surface coating on demoulding forces in replicating nano-scale surface structures. Especially, nano bead-like texturing and nano pillars on the tools' surfaces did not increase the demoulding forces to the levels witnessed on uncoated tooling surfaces, and the aC:H coatings remained a dominant factor in determining the tool performance. The carried out proof of concept study showed that the applied surface texturing method can be considered as an alternative to existing techniques for surface structuring and at the same time to reduce demoulding forces.

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1. Introduction

Polymer replication technologies allow the manufacture of a large variety of structured surfaces incorporating functional features within the micro to nano metre range. The industrial application potential of using replication processes to commercialised products enabled by micro and nano technologies is enormous, and the current technological state-of-the-art highlights both industry and research “push” for process optimization to enhance the replication fidelity (RF) at all scales [1]. The structuring of surfaces with micro and nano scale features also introduces additional functional characteristics such as anti-reflectivity, modified surface adhesion and friction forces, self-cleaning, water repulsion and large scale enhancement of cell attachment and alignment [2–5].

Micro injection moulding (μ -IM) enables a relatively low cost transfer of micro and nano metre scale structures from precision mould masters to moulded polymeric products [6]. The replication capabilities are limited to the surface geometry of the mould master and therefore the tool-making technologies are the gateway to realising successfully the replication of micro and nano surface structures.

The manufacture of mould tools that incorporate micro and nano features requires the capabilities of complementary micro/nano machining and structuring technologies such as micro electrical discharge machining (μ EDM), laser ablation, micro milling and focused ion beam (FIB) machining [7–9] to be combined innovatively. However, micro and nano structured surfaces on mould masters can be produced not only by these so called top-down structuring technologies but also by employing bottom-up technologies, such as beads and polymer based self-assembly technologies [10]. For example, surface functionalisation was successfully achieved by post processing the mould masters and thus depositing surface micro/nano features [1].

In μ -IM the optimisation of the filling and packing stages of the process is critical for achieving high RF and generally part filling requires high pressure (P) and temperature (T) [11–14]. One negative effect of fully filled and packed polymer parts is that it can increase the resistance in ejecting them from the mould during the demoulding stage of the process [15]. Especially, during the demoulding stage, part-mould ejection forces can cause stress marks, deformation, fracture and stretching of the polymer structures [16]. In the IM literature, ejection forces (F^e) are defined as the friction at the tool–polymer interface and they can be determined based on the part core surface area (A_c), the coefficient of friction of the moulded polymer (μ) [ISO 8295] and the determination of moulding contact pressure (P_A) [ISO 294-4] [17,18]. It is well known that the level to which the part is filled has a

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direct effect on the tool–polymer interface and thus on the friction between them. The polymers' coefficient of thermal expansion in general is one or two orders of magnitude higher than that of the mould material. Thus, a entirely packed volume of polymer in a cavity will lead to a complete fill of the surface features but at the same time any relatively high aspect ratio features on the mould surface will make demoulding difficult [19]. The mould surface finish is expected to have a significant effect on F^e ; however, the conducted research is not conclusive. In particular, it was reported that F^e increased both with the increase of the tool surface roughness and with the use of highly polished surfaces [20]. The necessity to improve RF of high surface to volume ratio (SV_R) and high aspect ratio micro and nano features in μ -IM calls for a decrease of part F^e and the need to maintain tool performance at an optimum level.

In this paper, the deposition of a amorphous hydrogenated carbon (aC:H) coating on mould surfaces that is textured incorporating nano beads and high aspect ratio nano pillar structures is investigated. The proof of concept replication of functional nano structured surfaces together with the influence of the aC:H surface treatment on the demoulding behaviour of parts with micro and nano features is reported. The paper is organised as follows. The next section reviews surface treatment technologies utilised in tool-making and the functionalities that can be introduced by applying them. Then, the experimental set-up and the test tool used to investigate the effects of the μ -IM process on textured aC:H coated mould tools are described. Finally, the experimental results are presented and the capabilities of this novel surface treatment of μ -IM tools is analysed.

2. Tooling surface technologies

Traditionally, improving the wear resistance of moulding surfaces was achieved through the applications of advanced steels, ranging from ingot cast martensitic matrix steels to advanced powder metallurgy tool steels with a high content of hard carbide particles [21]. However, more recently coating technologies for surface structuring/engineering have been applied to increase wear resistance and also to functionalised surfaces [15,22–24].

Structured surfaces are defined as 'surfaces with a deterministic pattern of usually high aspect ratio geometric features designed to give a specific function' [25], while engineered surfaces are defined as surfaces where the manufacturing process is optimised to generate variation in geometry and/or near surface material properties and thus obtaining a specific function [25]. Different manufacturing technologies can be applied to structure and engineer surfaces and they include a broad range of processes, e.g. sand blasting, innovative grinding systems, focus ion beam, nano-imprint lithography, chemical texturing and laser machining [22,23,26,27]. New innovative methods for introducing surface modifications that have been inspired by biomimetics and/or chemical methods of self assembly have been developed recently. By applying such methods it is possible to create homogeneous and non-homogenous patterns, topography and depressions on otherwise planar surfaces [2,24,26].

2.1. Structured surfaces

The replication of micro/nano structured surfaces was investigated by Worgull et al. to study differences in the wetting behaviours of patterned masters. The research demonstrated that surface structuring can decrease the wettability of PMMA substrates, e.g. the contact angle increases from 87° to 107°, when comparing with unstructured and structured surfaces respectively [28]. The structuring of surfaces with pillars' and grooves' arrays can be used in bio-sensing applications for cell monitoring and also for studying growth effects in tissue engineering [29,30]. Other biological applications were investigated by Giselbrecht et al. where scaffolds were created with three dimensional cell adhesion patterns [31,32]. Biomimetics inspired surfaces

with micro and nano topographies mimicking the eye of a household fly and a shark skin were replicated in polypropylene (PP) by micro injection moulding [2]. Recent studies have also demonstrated typical nano hair gecko-like geometrical arrangements on surfaces that can withstand shear forces of up to two times higher than surfaces without them [33].

2.2. Coated surfaces

Diamond like carbon (DLC) coatings belonged to the amorphous hydrogenated carbon (aC:H) group and the coating of plastic injection moulding inserts with DLC coatings improves the replication performance of the micro and nano structured masters [15]. This is achieved through a combination of superior tribological and mechanical properties such as low friction, low wear and high hardness [34–36] when compared to traditional tool materials. The low friction coefficient and low wear can be explained with the high ratio between the hardness and Young's modulus and the low ratio between the surface energy and hardness [37]. Typically, the surface treatment of inserts using pulsed laser deposition (PLD) of DLC coatings yields surface hardness of up to 70 GPa with friction coefficients in the range of 0.05–0.2, an order of magnitude lower than that of ceramic coatings [38]. Further investigations of DLC coatings where a special attention was paid to the inhibiting role of gas–surface interactions, showed that duty cycles with control variables of time and speed resulted in super low friction coefficients of 0.003–0.008 [39]. Further applications of DLC can be found in various fields such as magnetic storage media, sliding bearings, injection pumps, cutting and forming tools, data storage systems and anti-thrombus coatings [40–42]. It is important to stress that the application of DLC coatings is not limited to providing enhancement in mechanical properties but also a coating topography can be engineered to increase the surface functionality for applications such as anti-biofouling templates. This is achieved due to their extreme hydrophobicity (static contact angle higher than 160°) owing to low surface energy (24.2 mN/m) and dual-roughness structures' properties of the coatings [43].

In the research conducted by Saha et al. the effect of surface properties of micro structured masters on the hot embossing process was investigated by using nitrogen (N) and nickel (Ni) doped diamond-like carbon (N:DLC:Ni) coated and uncoated silicon (Si) micro moulds [44]. The results demonstrated that even with high friction and adhesion characteristics of this replication process the N:DLC:Ni coated Si masters successfully increased the mould lifetime by 3–18 times when compared against uncoated moulds. Bremond et al. studied the tribological behaviour of DLC-coated 100C6 when subjected to a temperature increase from a room temperature to 400 °C. The DLC coatings belonged to the amorphous hydrogenated carbon coatings' group. The results have shown that when used at temperatures higher than 200 °C the coating damage increases significantly due to stiffness reduction of the 100C6-steel substrate [45].

Another application for DLC coatings was investigated by Avelar-Batista et al. [46]. The objective of the research was to determine the DLC tribological performance when a Ti6Al4V alloy substrate was pre-treated with triode plasma nitriding to improve hardness. DLC and multilayered TiN/DLC, CrN/DLC CrAlN/DLC coatings were deposited onto 'standard' and plasma nitrided Ti6Al4V substrates. It was concluded that triode plasma nitriding had increased the load-bearing capacity of the coating/substrate system, as higher critical adhesion loads were recorded for DLC coatings of plasma nitrided Ti6Al4V substrates. In addition, pre-treatment also reduced the wear rate of the DLC coating/substrate system.

Sasaki et al. investigated the ejection force (F^e) as a function of surface roughness and surface coatings [47]. The experimental results demonstrated that when moulding PP and PET a reduced F^e and product deformation were achieved when the surface roughness of the inserts was in the range of Ra 0.212. The PMMA samples required a

lower F^c when surface roughness was Ra 0.092. Also, the results showed that any F^c reductions were depended both on the optimised surface roughness and the polymer material used in the moulding trials. In addition, it was concluded that the PVD WC/C carbon coating was the most effective in reducing F^c [47].

In micro injection moulding, large surface area to volume ratios are typical and result in high adhesion forces between mouldings and the tool surfaces [44]. However, it was reported that by treating the mould surfaces with DLC coatings when moulding PC and ABS polymers a F^c reduction of 40% and 16%, respectively, was achieved in comparison with untreated surfaces [15]. The benefits from using DLC coatings in micro replication processes are proven [44]. Therefore, it is important to continue researching the effects of the latest advances in the DLC coating technology when applying them innovatively in tool making and thus improving parts' functionality and quality. In this paper, the deposition of a novel structured aC:H coating on tool surfaces that incorporate surface engineered nano beads and high aspect ratio nano pillar structures is investigated. In particular, the wear resistance of this aC:H coating and the feasibility of replicating functional nano surfaces are studied.

3. Experimental set-up

3.1. Part design and tool manufacture

The part design used in this study is a 15 mm × 20 mm × 1 mm microfluidic platform shown in Fig. 1a. The system design includes features commonly found in microfluidic components such as reservoirs, channels and waste compartments. The cross section of the channels is 200 × 200 μm. Table 1 provides further information about the part design with and without its micro features. In particular, SV_R is 15.7% higher for the design that includes micro features.

The tool insert was manufactured in Uddeholm Stavax ESR SS 304 grade. It's a stainless steel for small and medium mould inserts and cores, and ideally suited for micro moulds. The steel was machined by micro milling. A draught angle of 1° was applied to all features, and the surface roughness achieved on the tool was Ra 0.30. A single 3 mm diameter pin was positioned at the centre of the part. Subsequently, an ejector system, tool 'fixed' and 'moving' halves were assembled to a primary mould tool.

3.2. aC:H coating

In this investigation, the surface treatment was carried out by depositing an a-C:H onto the tooling surface. The coating was performed in a low frequency PECVD reactor with a capacitively coupled electrode configuration. The lower electrode served as an $\varnothing = 250$ mm

Table 1
Part design characteristics.

Design properties	Design with micro features	Design without features
Volume	3.10 mm ³	3.37 mm ³
Surface area	8.33 mm ²	7.62 mm ²
SV_R	2.68	2.26

substrate holder powered via a 40 kHz transmitter. The distance between the two electrodes was kept constant at 200 mm while the vacuum chamber was pumped down to a base pressure ranging from 2×10^{-4} to 4×10^{-4} Pa. During the deposition process the working pressure was 4 Pa and the bias voltage of the substrates was –650 V, and the parameters were optimised based on research conducted on the characterisation of DLC deposition [48]. Prior to the coating, the substrates were ultrasonically cleaned in acetone and rinsed with ethanol before being dried and placed into the PECVD chamber. Then, argon and hydrogen plasma etching was performed at 5 Pa and 300 W for 20 min. The floating substrates' temperature (plasma power dependent) was below 150 °C. Cyclohexane (C₆H₁₂) diluted with hydrogen was used, as a gas precursor for aC:H film deposition. The hardness of DLC is high compared to the steel substrate, and to improve adhesion a buffer silicon doped aC:H layer was developed jointly with the amorphous hydrogenated carbon (aC:H) to insure the mechanical cohesion on steel substrates. In Particular, a hydrogenated silicon carbide bonding layer, 0.4 μm thick, was deposited on the substrate using a plasma of tetramethylsilane (Si(CH₃)₄) and argon. The method is based on work of C. Chouquet et al. [49,50]. The coated tool insert is shown in Fig. 1b. The structural and mechanical properties of the deposited aC:H film were studied previously [15,49,50] and are summarised in Table 2.

3.3. Surface texturing

A novel method was applied to texture the aC:H coating. Especially, the tool surface was textured by using a sacrificial hard mask of silica beads to perform an anisotropic etching of the thin aC:H coating. The precise control and reproducibility of the process resulted in a high aspect ratio texturing due to a more than 20 times high etching rate of the aC:H coating than the SiO₂ beads under O₂ plasma exposure. A schematic representation of the process steps is shown in Fig. 2. In particular, the process includes the following three steps:

1. Deposition of SiO₂ nano beads on the DLC coating using the Langmuir-Blodgett process.

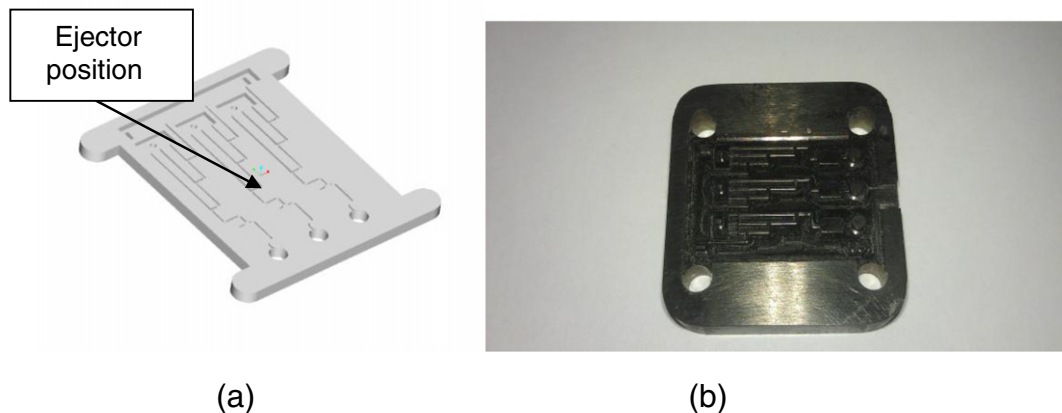


Fig. 1. (a) Micro fluidics part, (b) DLC coated tool insert.

Table 2
Structural and mechanical properties of aC:H film.

Properties [15,49,50]	aC:H
Coating thickness (μm)	1
Hardness (GPa)	21 ± 2
Young modulus (GPa)	160 ± 9
Residual stress (GPa)	1.2 ± 0.1
Friction coefficient (μ)	0.15
Wear rate ($\text{mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$)	3×10^{-8}
Csp ² /Csp ³ (RMN)	3
Total H content (%)	25 ± 2

- Etching the DLC layer under O₂ plasma exposure. The etching rate is approximately 0.7 nm/s under the applied conditions, O₂ flow rate of 80 sccm, power – 10 W, pressure – 3 Pa and bias – 320 V, and thus the depth is controlled by monitoring the exposure time.
- Removal of SiO₂ nano beads in DI water with ultrasounds at room temperature.

As can be seen in Fig. 3 nano beads can be deposited with different densities, high and medium populations. Fig. 4 depicts the flexibility of the surface texturing process, especially in engineering DLC pillars of different group sizes and also in being capable of producing single high aspect ratio pillars that have a bead-like topography.

3.4. Demoulding force measurements

In this study, variations in F^e during the μ -IM process were assessed using a novel condition monitoring system developed for μ -IM. To carry out the measurements a Kistler 9211B miniature force sensor positioned behind the ejector pin, as shown in Fig. 5, measures F^e by means of the ejector movement. When the ejector assembly moves forward during the ejection cycle the part is removed from the cavity while the sensor is subjected to a mechanical load that generates an electric potential. The sensors' piezoelectric charge is then converted using an ICAM charge amplifier into an output voltage proportional to the applied mechanical force. The output signal is monitored using a National Instruments NI 9205 16-bit module and recorded into a PC using a National Instruments data acquisition unit. Then, the data is analysed by employing the National Instruments Labview 8 software. Finally, the key metrics used to compare the ejection force curves are then calculated using the Matlab™ software. The focus of this research is on the behaviour of the structured aC:H coating during the μ -IM process, in particular the maximum demoulding forces (F_{Max}^e) that parts are exposed to. The recorded F^e curve in Fig. 6 depicts the demoulding stage

of the injection moulding cycle and F_{max}^e that the part is subjected to. The demoulding force was calculated using the following equation:

$$F = \frac{\text{Output}(v) \times 99.5(\text{pC})}{E_f} \quad (1)$$

where: E_f is the force sensitivity, –4.4 pC/N.

To compare the ejection force curves the software Matlab™ was utilised to calculate F_{max}^e , which represents the maximum demoulding force during the demoulding stage of the cycle at time (t):

$$F_{max}^e = F^e(t_{max}) = \max(F^e(t)) \quad (2)$$

where t_{max}^e represents the time when the demoulding force reaches its maximum value.

3.5. μ -IM experiments

Due to the fact that the demoulding performance relies heavily on the T control during injection, the effects of barrel temperature (T_b) and mould temperature (T_m) are investigated. To ensure that the polymer has solidified sufficiently to facilitate the part ejection without deformation, the cooling time after part filling (t_c) and a time delay for controlling the ejection time (t_e) were introduced. The t_e parameter can be defined as the set time for the polymer to cool down before the demoulding stage starts. Whereas, the effect of t_e can be witnessed when the mould opens and the part is partially exposed to ambient temperature and the delay facilitates additional cooling. In previous research [15] the theoretical best set of processing parameters in terms of a reduced demoulding force was established (Table 3). Based on these results the four main control factors identified are used in this research. The response of a tool surface that is treated and untreated was investigated by analysing the F_{Max}^e during demoulding. Given that two tool surfaces, untreated, and aC:H coated, are investigated and a total of 20 trials were carried out.

4. Analysis of the results

4.1. Influence of the nano structured coating on the demoulding force

In this study, the set of processing parameters was selected to ensure that the experimental results were obtained when low demoulding forces were applied. To investigate the influence of the nano textured coating, ten mouldings were produced using textured inserts and the demoulding forces were recorded. The insert was then stripped from its coating using oxygen plasma etching and the moulding experiment repeated. For each trial, the effects of the applied surface treatments on F_{max}^e were analysed and then based on

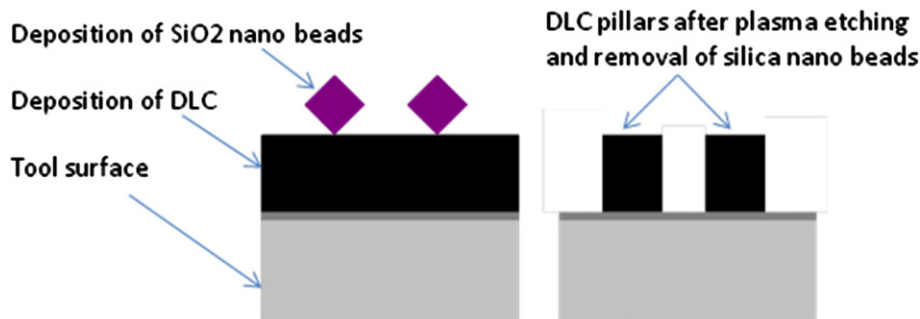


Fig. 2. Schematic representation of the process steps for texturing DLC coatings, SiO₂ nano bead deposition over a SiCH/DLC stack followed by O₂ plasma etching of the coating and nano beads removal in an ultrasonic bath with DI water.

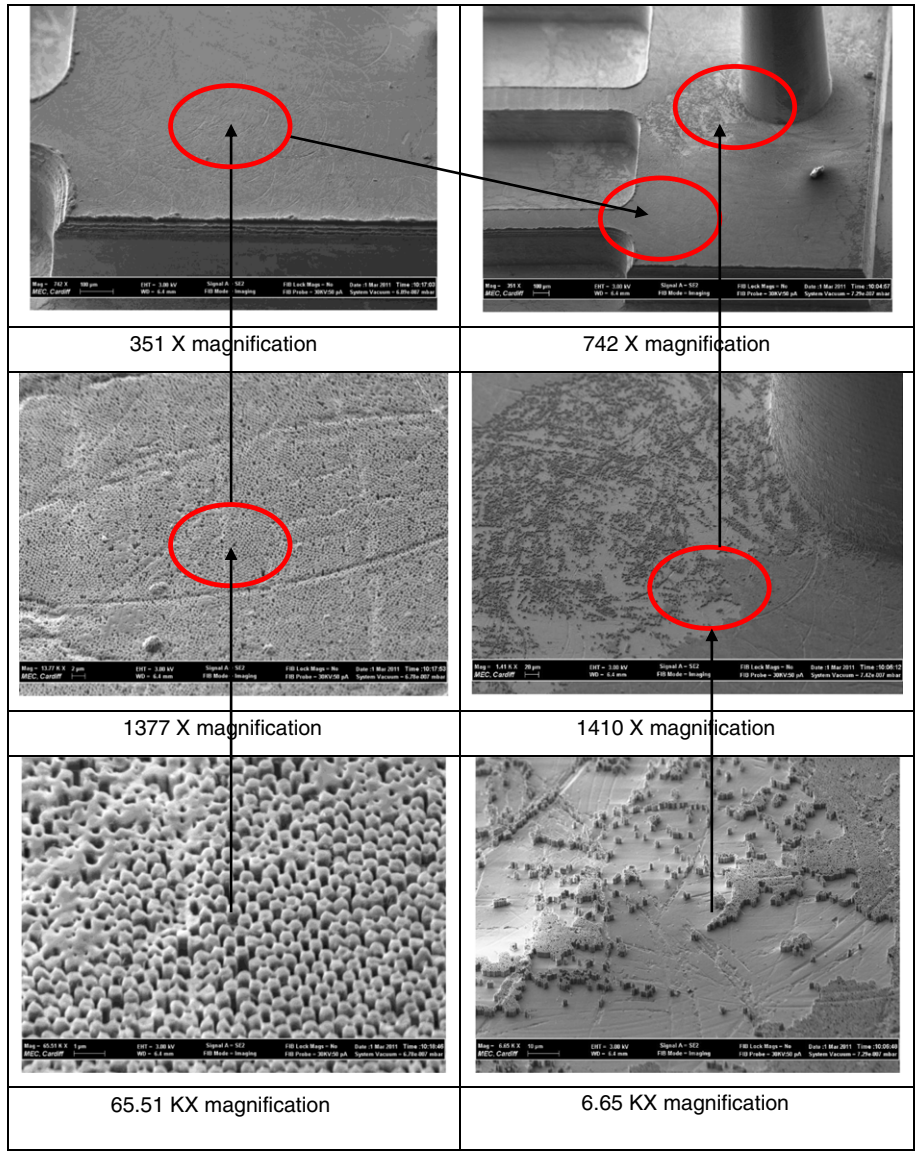


Fig. 3. aC:H surface treatment with high and medium populations of nano beads.

the obtained results the mean values were calculated for both tools, as shown in Fig. 7. Immediately it was apparent that the F^p curves for the treated and untreated tools were different (Fig. 7). The histogram

of average results in Fig. 8 clearly shows that the tool with the nano structured coating has F^p_{max} that is lower than that obtained using the untreated one. In particular, an average reduction of 15.8% was

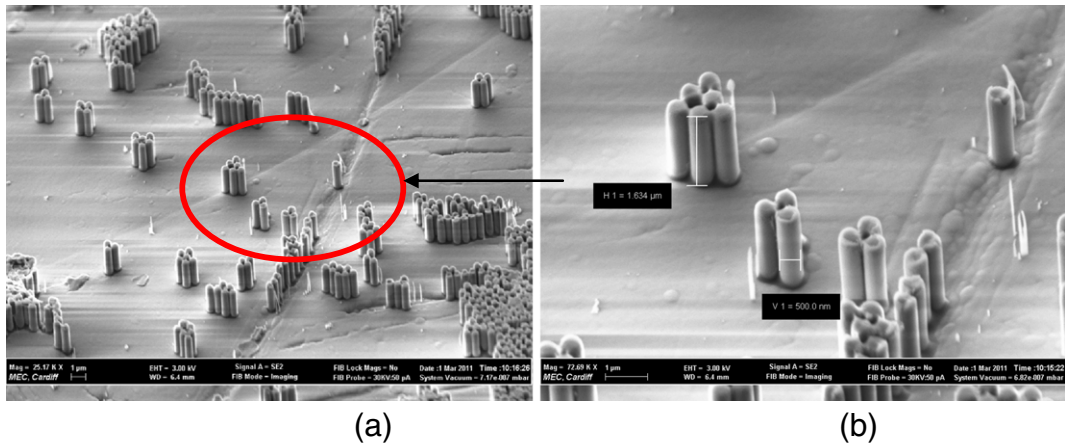


Fig. 4. Low populations of high aspect ratio nano pillars, (a) 25.17 KX and (b) 72.69 KX magnifications, respectively.

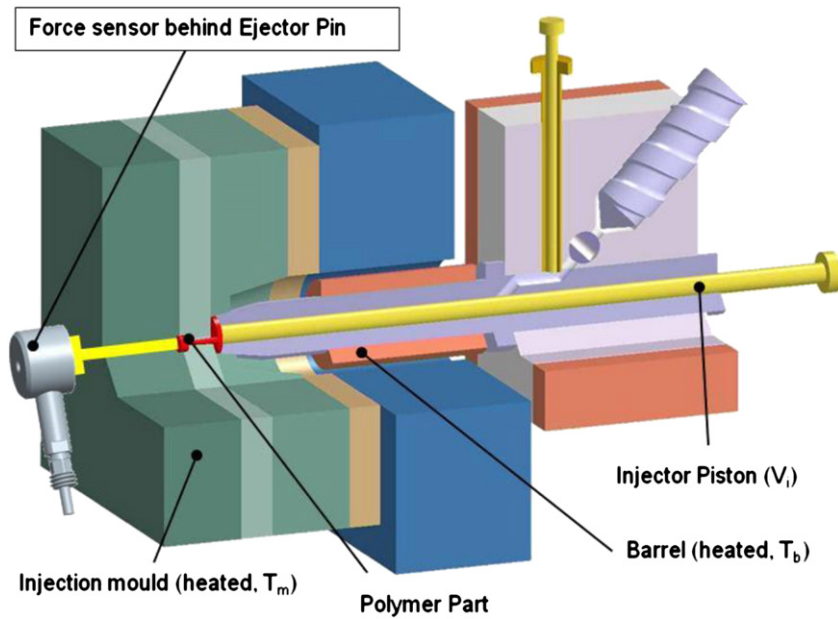
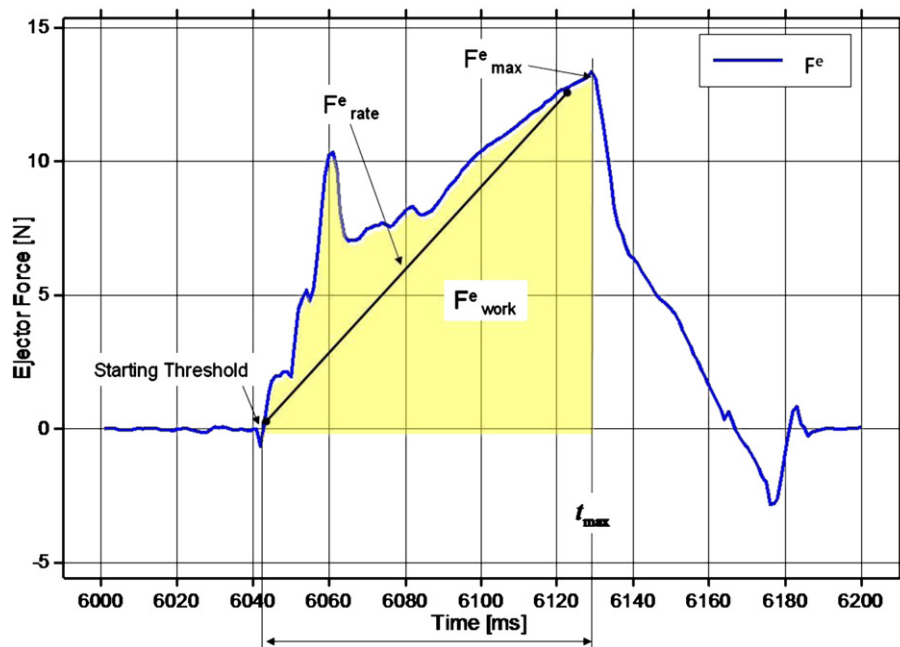
Fig. 5. F^e measurement positions.

Fig. 6. The demoulding force curve over time.

obtained (Table 4). Thus, the aC:H treated tools show a significant reduction in demoulding forces in μ -IM. In addition, these results quantify that even with a nano structured surface that should certainly lead to an increase of the polymer-tool adhesion, the coating is still dominant in reducing part-mould forces during the ejection cycle.

Table 3
The theoretical best set of processing parameters [15].

T_b [°C]	T_m [°C]	t_c [s]	t_e [s]
230	40	1	10

4.2. Replication of the textured aC:H coated surfaces

The inspection of the coated insert using scanning electron microscope imaging, as shown in Fig. 9, revealed that over the majority of insert surfaces, the aC:H texturing produced densely populated areas of pillars with 0.5 μm diameter and 1.6 μm height. However, as it can be observed in Fig. 10, the population of pillars is reduced and irregular in some areas. The reduced and irregular nano beads' coverage was low in these regions as a direct result of a liquid meniscus breakage during the Langmuir–Blodgett process. To determine if the nano textured aC:H coating could withstand the μ -IM process and an initial batch of 100 components were produced in PP. The resistance of the textured coating to any delamination during the μ -IM

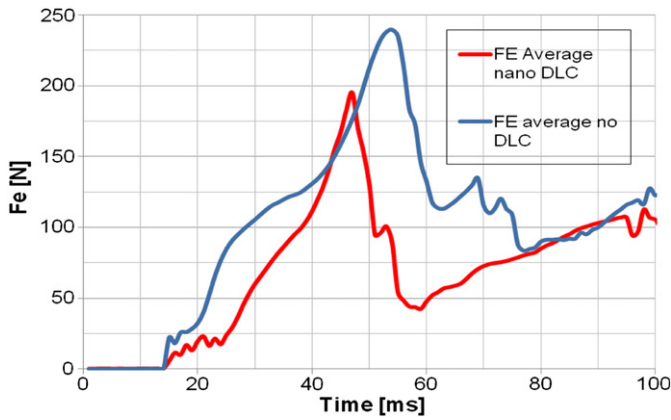


Fig. 7. Typical demoulding curves for treated and untreated tools.

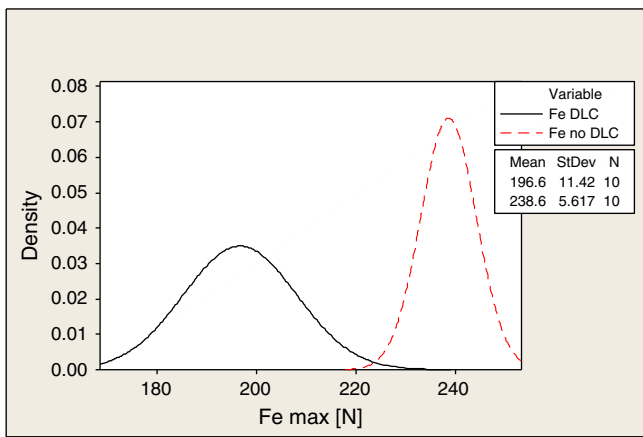


Fig. 8. Histogram of average results for the treated and untreated tool.

Table 4
 F^e_{max} results.

F^e_{max}	Max	Min	Average
Treated tool	208.0	178.1	196.6
Untreated tool	244.9	230.8	233.5
Reduction [%]	15.07	-22.8	-15.8

process was high. No visible signs of the coating deterioration were observed after the moulding trials either in the high and low population areas as illustrated in Figs. 9b and 10b.

To facilitate the surface replication process the μ -IM settings were optimised in order to reduce the demoulding forces. The bead-like textured coating on the tool surfaces was replicated as depicted in Fig. 11. Regarding the areas with an irregular texturing the replication results were more varied as can be seen in Fig. 11b. Trenches with bead-like textured bottoms can be observed while the untreated areas resulted in flat “plateaus”. Also, it can be observed in Fig. 11c that single and small groups of pillars were replicated as holes and/or small trenches in the PP mouldings. Following a further inspection it can be seen that the holes are not straight but angled. This is due to the ejection angle of the part. With only one ejector pin utilised, the line of draw is not sufficiently balanced for a parallel part removal from the cavity. The imbalance results in an additional stress on the DLC pillars but in spite of this most pillars remained intact. This is another proof about the extremely high strength of the pillars. Nevertheless it is important to note that one isolated pillar in the low density areas had broken from the tool surface and was trapped into one of the part mouldings (Fig. 11d).

5. Conclusions

In this research an experimental study is reported that investigates the performance of a textured aC:H coating used in a μ -IM tooling application. A novel surface patterning method is employed to produce sub-micron texturing on tooling cavities. In particular, the investigation focused on the effects of the textured coating on resulting demoulding forces when replicating a microfluidic component. Using a novel condition monitoring system that can capture data accurately during demoulding cycles, the performance of treated and untreated tooling surfaces was compared. In addition, an empirical data was collected to assess the ability of the textured surface to withstand recurrent loads during the moulding cycles and the capability of the process to replicate the surface structures and nano pillars. The following conclusions can be made based on the obtained results:

1. In spite of treating the tooling cavities with a textured aC:H coating a reduction of F^e by 15.8% was obtained in comparison with the results on untreated surfaces. In particular, the nano bead-like texturing and nano pillars on the tool surfaces did not increase the demoulding forces to levels witnessed on uncoated tooling surfaces. Thus, the significant reductions of friction coefficients that are associated with the aC:H coatings remain a dominant factor in determining the tool performance.

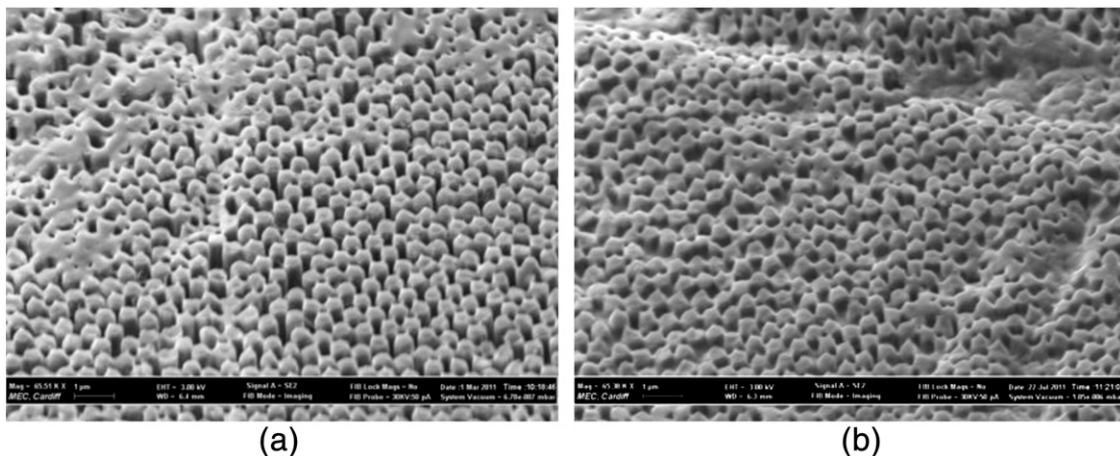


Fig. 9. A tool surface that is highly populated with pillars before (a) and after (b) 100 PP mouldings.

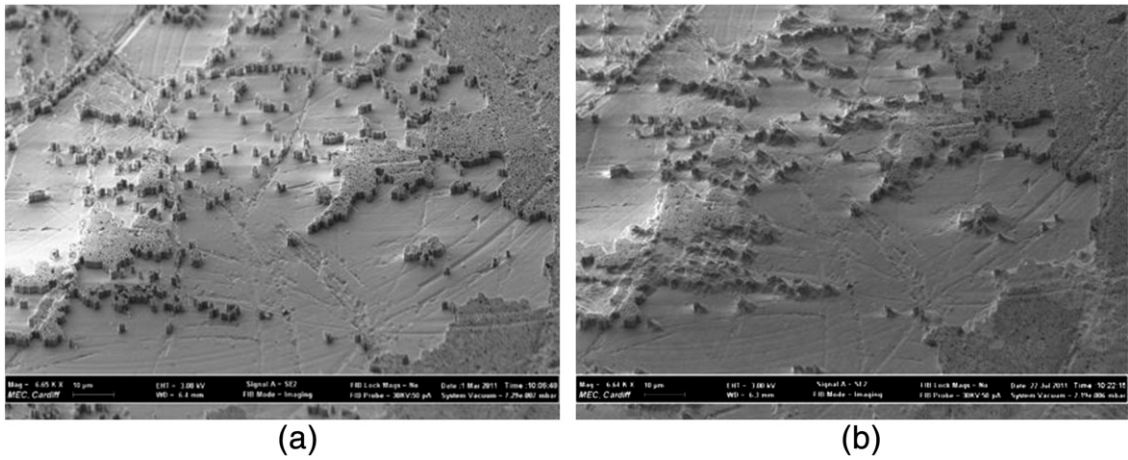


Fig. 10. A tool surface that is irregularly populated with pillars before (a) and after (b) 100 PP mouldings.

2. The aC:H coatings showed a very strong adhesion to the tool surfaces. No deterioration or delamination of the textured coatings was observed both on the highly covered areas and the irregularly ones. Single high aspect ratio pillars withstand over 100 cycles of filling and packing the tool cavities and also the demoulding forces. It was observed that only one pillar was broken as a result

of demoulding. Therefore, to extend the “life” of such high aspect ratio structures, it is critical to design and balance accurately the ejection system and the part demoulding process.

3. The inspection of the moulded PP components showed that the nano bead-like texturing was replicated fully together with some small holes from the replicated nano pillars. The μ -IM settings

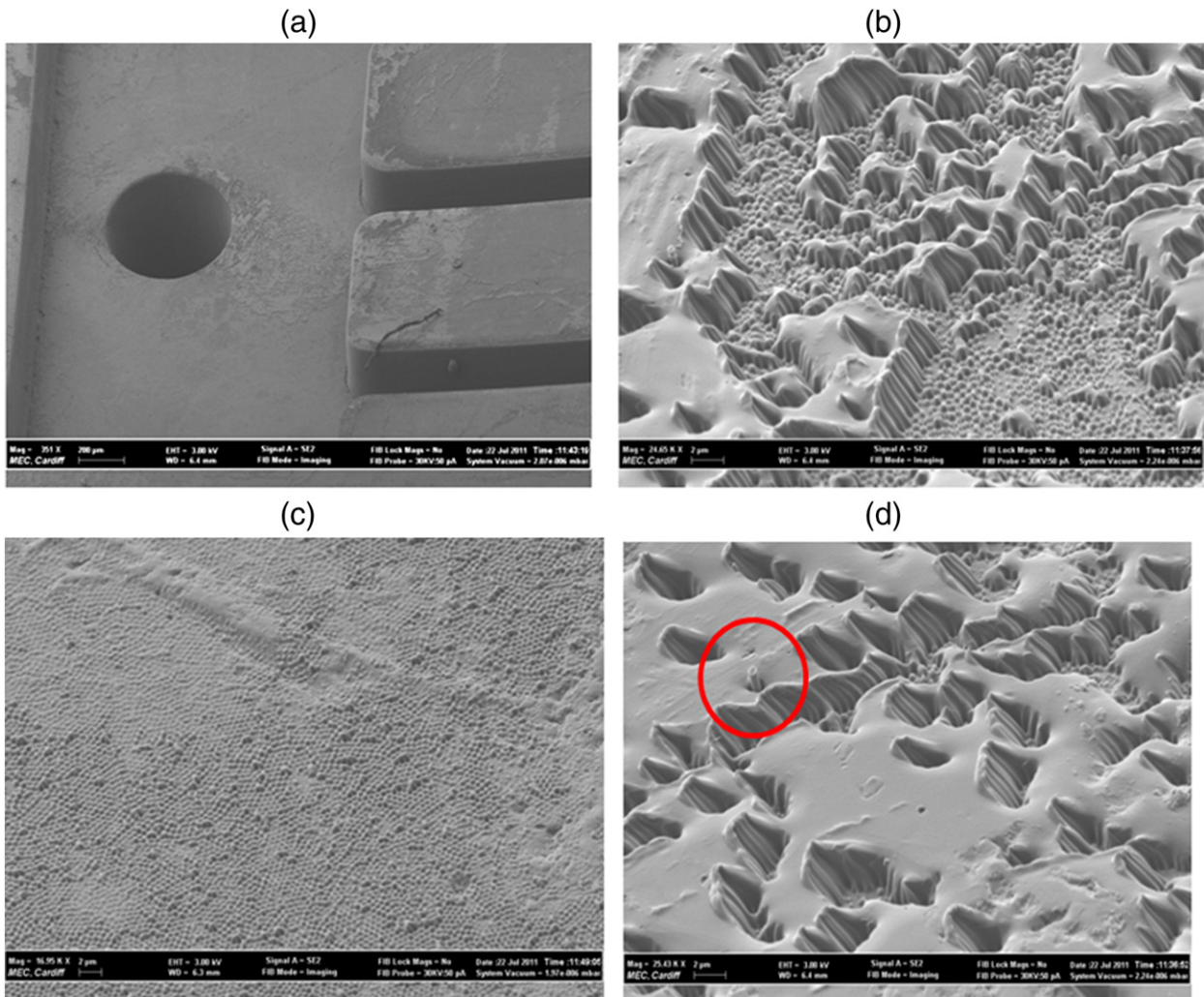


Fig. 11. (a) Irregularly populated nano beads on a PP moulding; (b) an irregular aC:H texture replicated onto a PP moulding. (c) The texturing produced onto a PP moulding with a tool surface that was highly populated with pillars. (d) A nano pillar trapped into a PP moulding.

were not optimised for improving the replication performance but only for reducing the demoulding forces. Further research is necessary to determine the best set of processing parameters and thus improving replication fidelity while maintaining and even reducing the demoulding forces.

Finally, the carried out feasibility study showed that the applied surface texturing method can be considered as an alternative to existing techniques for surface structuring and engineering. The research has shown that the aC:H coatings are robust, and advantageous in terms of reducing the inherent forces during demoulding. Further process optimisation is necessary to widen the application area of this novel surface treatment technology, in particular to improve the uniformity of the textured aC:H coatings and also to make a possible selective texturing or the deposition of single and multiple features with a high degree of accuracy.

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