



# Three-level hierarchical micro/nanostructures on biopolymers by injection moulding using low-cost polymeric inlays

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Received: 5 August 2022 / Accepted: 15 October 2022 / Published online: 1 December 2022  
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## Abstract

The industrial interest in the patterning of polymeric surfaces at the micro/nanoscale to include new functionalities has considerably increased during the last years. Hierarchical organization of micro/nanometric surface textures yields enhanced functional properties such as hydrophobicity, hydrophilicity, antibacterial activity, and optical or chromatic effects to cite some. While high accuracy methods to pattern hierarchical surfaces at the nanoscale have been developed, only some of them have been applied for high volume manufacturing with limited success, mainly because they rely on the use of expensive machinery and moulds or complicated inserts. Therefore, a method using low cost recyclable tooling and process conditions applicable to high-volume manufacturing is currently missing. In this work, a scalable and low-cost method to replicate hierarchical micro/nanostructured surfaces on plastic films is presented, which can be latter used as inlays for injection moulded parts with standard processing conditions. This method is used to demonstrate the feasibility of replicating three level hierarchical micro/nano textured surfaces using recyclable bio-based polymers (of high relevancy in the current plastic pollution context) achieving replication ratios above 90%, comparing the replication results with those obtained in polypropylene. The presence of the micro/nanotextures substantially increases the contact angle of all the polymers tested, yielding values higher than 90° in all the cases. Also, various mechanical properties of the replicated parts for all the polymers injected are characterized one and thirty days after the samples were manufactured, showing fairly constant values. This highlights the validity of the replicated surfaces, regardless of the biopolymers special crystallization characteristics.

**Keywords** Micro/nanoscale structures · Hierarchical structures · Nano-imprint lithography · Injection moulding · Flexible inserts

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## 1 Introduction

It is well understood that the presence of microstructures and nanostructures in the inner structure and in the surface of materials can enhance their functional properties. Numerous examples of functionalities have been identified in the natural world [1, 2] and most of the scientific principles have been successfully elucidated. Some of the existing natural micro- and nanostructures are hierarchically organized. Such arrangement is commonly manifested at several dimensional levels, that might even present different chemical composition and it is responsible for a notable improvement of key physical properties, among which self-cleaning [3] or super-hydrophobicity [4]. Hierarchical micro- and nanostructures can be found in bones, wood, plant surfaces [4], or insect cuticles [2, 3] and wings [5].

Several methods have been developed to emulate these natural existing structures on the surface of polymeric parts, in view of improving their functionalities in diverse application fields. Most of them are based on laboratory-scale procedures. One of the most common methods is nanoimprint lithography (NIL). NIL is a high-resolution patterning technology based on replication of the surface features of a mould into a polymer material, by mechanically moulding or embossing, using the action of temperature or UV light crosslinking. While NIL was initially originated as a nanometric resolution lithographic method, able to compete with optical lithography, it evolved to a general method to create functional nanostructured surfaces for applications beyond microelectronics. Related NIL methods have been developed, including the use of soft stamps (Soft-NIL) [6], which is particularly well suited for large, and not necessary flat substrates NIL can be scaled up to provide high throughput replication by means of roll-to-roll techniques. Some examples of replication of multilevel hierarchical surfaces by NIL can be found in [7].

Amongst the common replication techniques on polymers, injection moulding constitutes one of the most important methods for mass production and gathers therefore the highest industrial interest due to its reduced cost per unit and possibilities of producing versatile shapes [8]. The main factors influencing the quality of the replication are the election of the injected polymer, the geometry of the mould and cavities to be filled, moulding temperature, melting temperature, injection & holding pressure, cooling time, and injection velocity as shown by numerous previous studies [9–12]. The most commonly used mould tool materials in injection moulding are metallic alloys. For example, Zhang et al. [13] used microinjection moulding with nickel inserts to study the filling behaviour of high aspect ratio microfeatures on a microfluidic flow cytometer chip by using short-shots. Blondiaux et al. [14] used microstructured and nanostructured steel inserts obtained by micromilling & etching processes to replicate a bio-diagnostic platform using polycarbonate material. Bhuyan et al. [15] used micromachined and hot water-treated aluminium to replicate hierarchical micro/nanostructures on polypropylene.

However, it is found that although metals are highly durable and practical to be used as mould materials because of the many available processes to structuring their surface, the maximum achievable filling ratios are limited due to their high thermal conductivities [16]. High thermal conductivities revert into a very quick formation of a frozen polymer layer once the molten polymer contacts the cold mould material, which impedes the further filling of the micro/nanostructure. That is why

advanced injection moulding techniques such as ICM and VIM (injection compression and variothermal injection moulding) or VICM (variothermal injection compression moulding) are required [17] when using metallic moulds to achieve optimal and consistent replication rates of the micro/nanostructures. These add considerable tooling costs to the injection process and additional relevant costs, such as energy consumption, due to the extended cycle times for ICM and VIM, have to be also considered when determining the overall process costs.

An alternative technique to obtain consistent replications of microstructures and nanostructures at lower tooling costs is the use of coated polymeric films as flexible mould tooling material. In this technique, the coated polymeric film acts as an effective thermal barrier that can significantly hinder heat transfer into the mould during the moulding process, which might keep the melt viscosity low (and therefore the melt flowing ability high) for longer time [18].

Stormonth-Darling et al. [19] used polymer films patterned by NIL as mould inlay material to replicate high-aspect ratio nanostructures on common thermoplastic polymers. In his research, a photoresist typical employed in microsystems technology (SU-8 3000 series) was coated onto a highly technical polymeric film (Kapton<sup>®</sup>) and subsequently coated with a fluorosilane—for easier part demoulding—allowing to replicate high-aspect ratio pillar-like nanostructures.

In terms of the replication of hierarchical structures on common thermoplastic materials using low-cost tooling, even though some relevant work has been carried out with two-level hierarchical micro/nanostructures [15, 20], to our knowledge, only few publications have been carried out combining three level hierarchical micro/nanostructures, low-cost recyclable tooling, and bio-based/biodegradable thermoplastic polymers. Therefore, further research is still needed to further reduce the cost of the tooling materials towards a potential industrialization of the technology, and also to extend the range of micro/nanostructured polymers to the family of the bio-based polymers, highly interesting [21] in the current plastic pollution and climate-change context for their bio-based origin and biodegradability.

Here, we present a method to replicate three level hierarchical micro/nanostructures on relevant bio-based and bio-degradable thermoplastic polymers using low cost and recyclable coated polymeric inlays. Additional to the quantification of the replication fidelity, we also evaluate the improvements in the contact angles experienced on the surface of the injected polymers (changing their wetting character from hydrophilic to hydrophobic), together with the comparison of the mechanical resistance of the micro/nano textured surfaces on the injected biopolymers with the non-textured ones.

## 2 Materials and methods

### 2.1 Fabrication of the Ormostamp® stamps

Figure 1 shows the overall fabrication process for obtaining the plastic inlay stamps. The process consists of 7 steps: (1) Fabrication of the silicon mould, which contains the pattern to be transferred to the final polymer sample. (2) Thermal NIL to transfer to a PMMA Film. (3) PDMS casting and (4) curing to obtain a replica of the PMMA film. (5) On this PDMS, Ormostamp® [22] is casted and (6) subsequently cured while slight pressure with a plastic foil is applied, which will form the inlay stamp to be used in injection moulding.

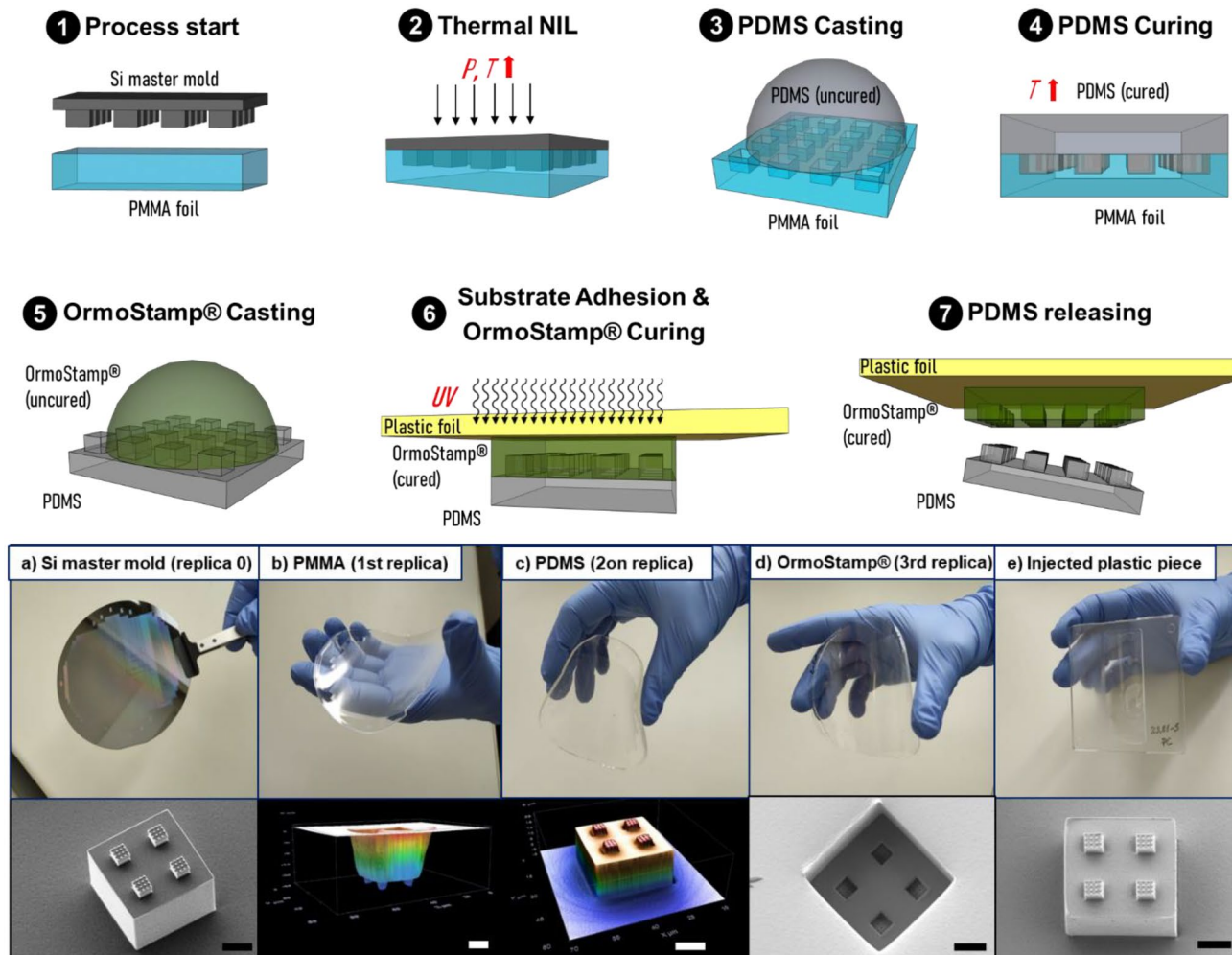
The process has two main advantages: (a) The PDMS film can be re-used many times, so that both Si master and

thermal NIL are preserved and (b) Ormostamp® can be easily released from the PDMS film much easier than from the silicon Stamp. It is worth to note that an odd number of replicas is needed to obtain the same polarity of features in the silicon stamp and in the final injected polymer part.

The details and main aspects of the stamp fabrication are presented in the supplementary information. The design of the hierarchical structure was performed in the framework of a previous study to analyze its influence on surface hydrophilicity [23].

### 2.2 Injection moulding

For the injection moulding tests, an *Engel complet E-motion 200/55* electric machine was used. The main processing parameters applied for each of the polymers injected are presented in Table 1.



**Fig. 1** (Top) Sketches of each individual process step of the method to obtain plastic inlay stamps from a silicon mould. (Bottom) Photographs (upper row) and micrographs (lower row) at each step of the process: silicon stamp (a), replicas (b, c), and a final polycarbonate

injected plastic piece (e). Micrographs images (a, d, e) are obtained by SEM while the micrographs of the images (b, c) are obtained by confocal microscopy. The scale bar is 10  $\mu\text{m}$  for all the images

**Table 1** Processing parameters used during the injection moulding trials

	PBS	PHB	PLA
Melt temperature (°C)	130	175	205
Mould temperature (°C)	20	20	20
Injection speed (mm/s)	50	60	60
Holding pressure (bar)	700	500	600
Holding pressure time (s)	5	5	5
Cooling time (s)	20	15	20

A photograph and an SEM image of the coated film are presented in Fig. 2. In order to hold the micro/nano textured films during the injection moulding process, a custom-designed injection mould insert was manufactured. The insert allowed the placement of the coated films interchangeably on the surface of the fixed half in the injection mould in order to carry out replication trials (see supplementary information).

The following biopolymers have been used in the injection moulding experiments:

- PLA BIO LM 99004 from *Ercros*,
- PBS PBI 003 from *Natureplast*
- PHB P304 from *Biomer*

In addition, PP C100 CA50 from *Ineos* was also used for comparison purposes. All the polymers were injection moulded according to the standard processing recommendations from each of the specific manufacturers, using moulds at room temperature in all cases.

## 2.3 Characterization

### 2.3.1 Degree of replication (%) obtained with confocal microscope and SEM images

For the morphological characterization of the injected samples a *Sensofar Plμ 2300* confocal microscope was used, and the images acquired were latter processed by the software *MountainsMap 5.1* of *Digital Surf*, and *Gwyddion*.

The main purpose of this characterization was the determination of the degree of replication, DR%:

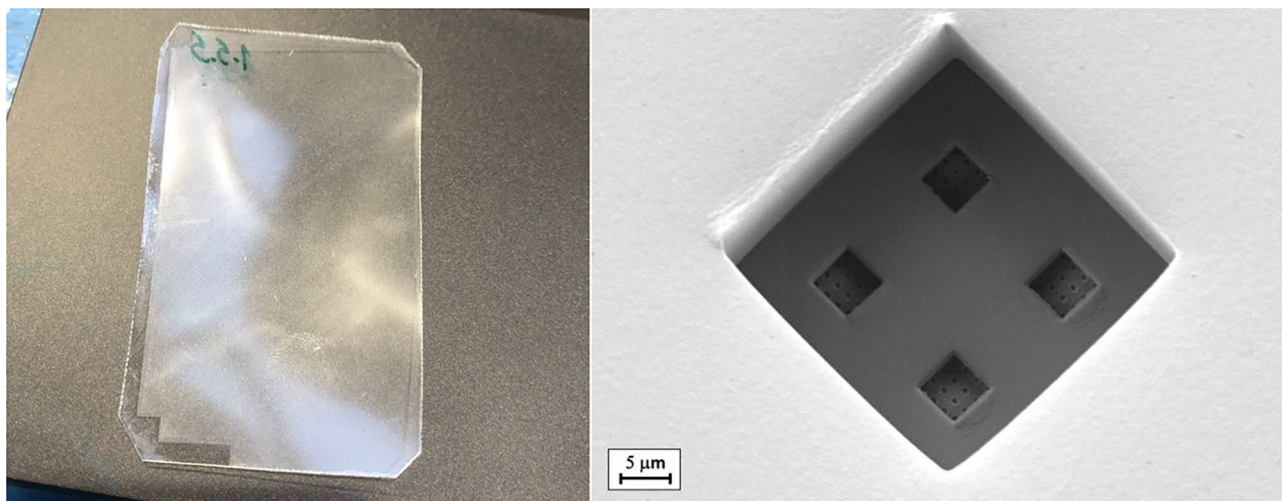
$$DR\% = h_f/d_c \times 100 \quad (1)$$

where  $h_f$  is the height of features in the polymeric sample and  $d_c$  is the depth of cavities in the coated polymeric inlay film.

The DR% parameter is also interesting to characterize the uniformity of the replicated structures on the injected polymer samples.

### 2.3.2 Water contact angle

Water contact angle measurements (WCA) of the Ormostamp<sup>®</sup>-coated PET films and the micro/nanostructured zones were obtained by means of a tensiometer (Dataphysics OCA 15 CE) and image processing software (SCA 2.0). Micro-droplets of 150 μL of deionized water were used. The contact angle values were obtained as the average of 5 measurements.



**Fig. 2** Details of the inlay stamp consisting of a micro/nano textured polymer-coated PET film (left) and SEM image detail of the 3-level hierarchical negative structures on it



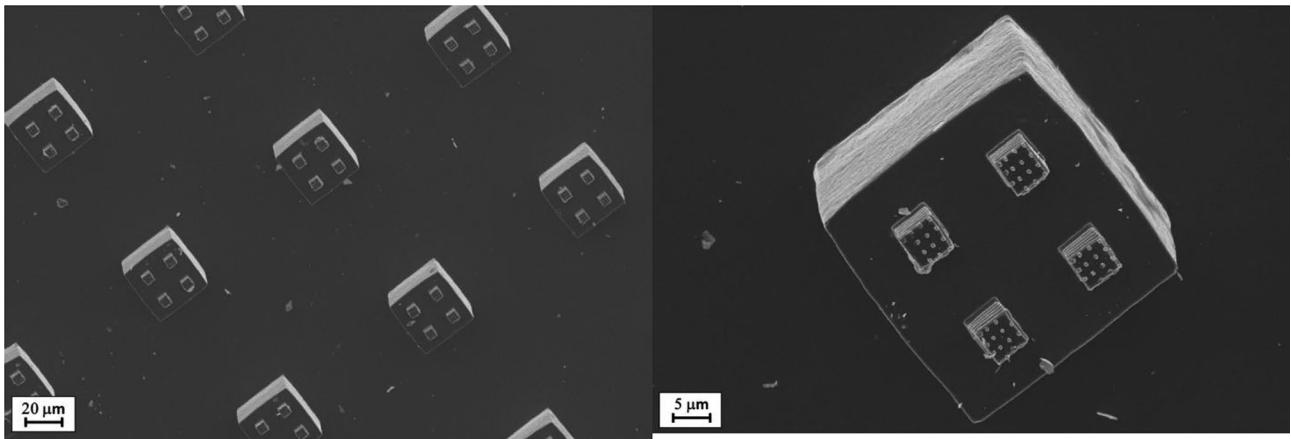


Fig. 3 PHB injected part. Hierarchical micro/nanotextured zone (left). Detailed single feature of the same PHB part (right)

### 2.3.3 Mechanical characterization-Scratch and flat punch micro tests

In order to evaluate the different scratch resistance and quantify the coefficient of friction (COF) of the micro/nano textured zones vs the non-textured ones on the injected polymer

samples, surface scratches were practiced at 0.5 N and 1.5 N load levels using a *Micro-combi indentation-Scratch Tester MHT (CSM)*. Also, *Flat Punch* indentation measurements using an AISI 52100 steel ball of 1 mm diameter were taken in order to evaluate the elastic storage modulus of the injected samples under compression in both textured and non-textured zones.

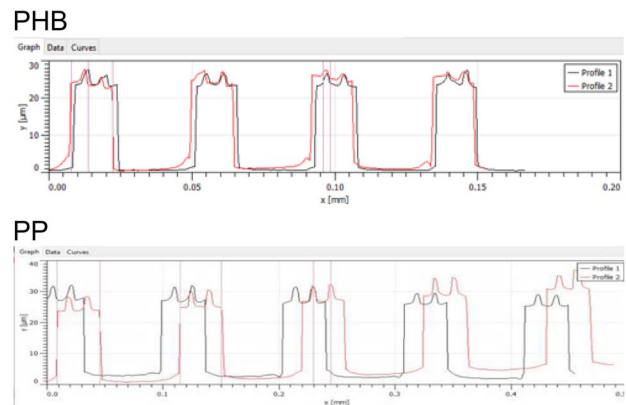
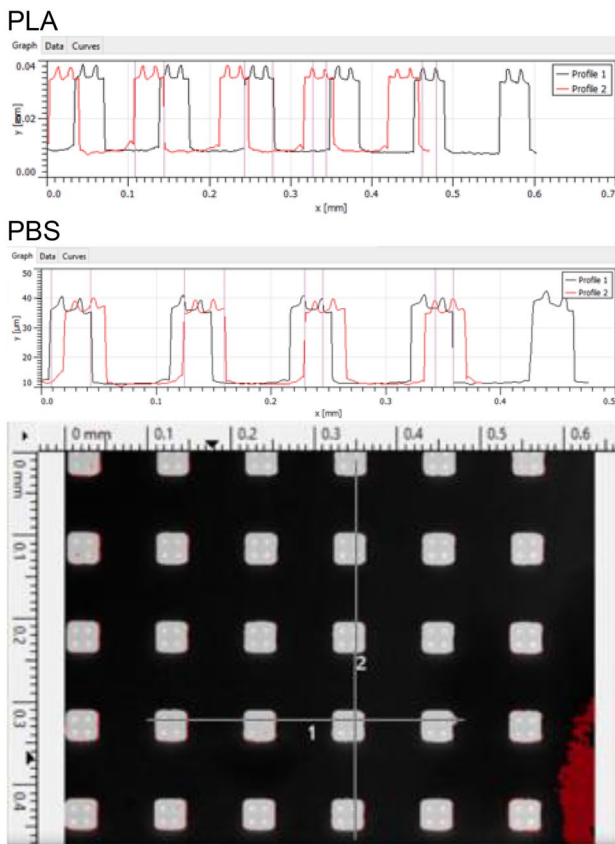


Fig. 4 Extracted confocal profiles (black line = horizontal profile across features/red line = vertical line along features) of the surface of the micro/nanotextured replicated polymeric parts via injection

moulding. The confocal optical image in the left presents the position of extraction of the horizontal (1) and vertical (2) profiles

**Table 2** Contact angle measurements

		CA °	SD
PLA	Non textured	82.28	±1.55
	Textured	105.13	±3.22
PHB	Non textured	53.73	±5.66
	Textured	95.37	±1.77
PBS	Non textured	80.31	±1.1
	Textured	93.62	±1.74
PP	Non textured	100.35	±1.39
	Textured	127.7	±1.96

Both mechanical measurements were taken 4 days after the injection moulding took place and also after 33 days, to check the time evolution of the surface mechanical properties. It is well known that the stiffness increases due to post-crystallization (also called cold crystallization) for some of the biopolymer types used [24, 25]. This increase would lead to a higher part brittleness and therefore an overall decrease of the part mechanical properties, which could affect the replicated micro/nano textures.

### 3 Results and discussion

#### 3.1 Quality of the replication

The patterns replicated achieved average degrees of replication (DR%) comprised between 90 and 110% for the three polymers. The percentages higher than 100% represent excessive micro/nano texture heights and therefore considerable stretching, most probably caused during the demoulding stage. Details of the overall uniformity on the micro/nanotextures and the replication of the three hierarchical levels can be observed in Fig. 3, and extracted confocal profiles of the injected samples are presented in Fig. 4.

It is remarkable that the degrees of replication are always around 100% when we compare the inlay film with the patterns in the silicon mould and also when we compare the patterns in the injected plastic parts with the patterns in the

in-lay film. The variability in the measurements ( $\pm 10\%$ ) are within the experimental error of the measurements.

In some of the injected samples, the micro/nano features presented an elongated and deformed shape (see supplementary information), which could have been caused by excessive adhesion between the film and the injected part due to an insufficient cooling time or to an excessive adhesion between the moulded part and the micro/nanotextured OrmoStamp<sup>®</sup>. This phenomenon took place occasionally for all the biopolymers tested, especially in the upper zone of the injected part, while it was more usual for the injected PP samples (injected at higher melt temperature as per manufacturer specifications). This could have been produced by the slightly higher temperature existing in the upper region of the plastic part with regards to the rest of it at the moment of demoulding.

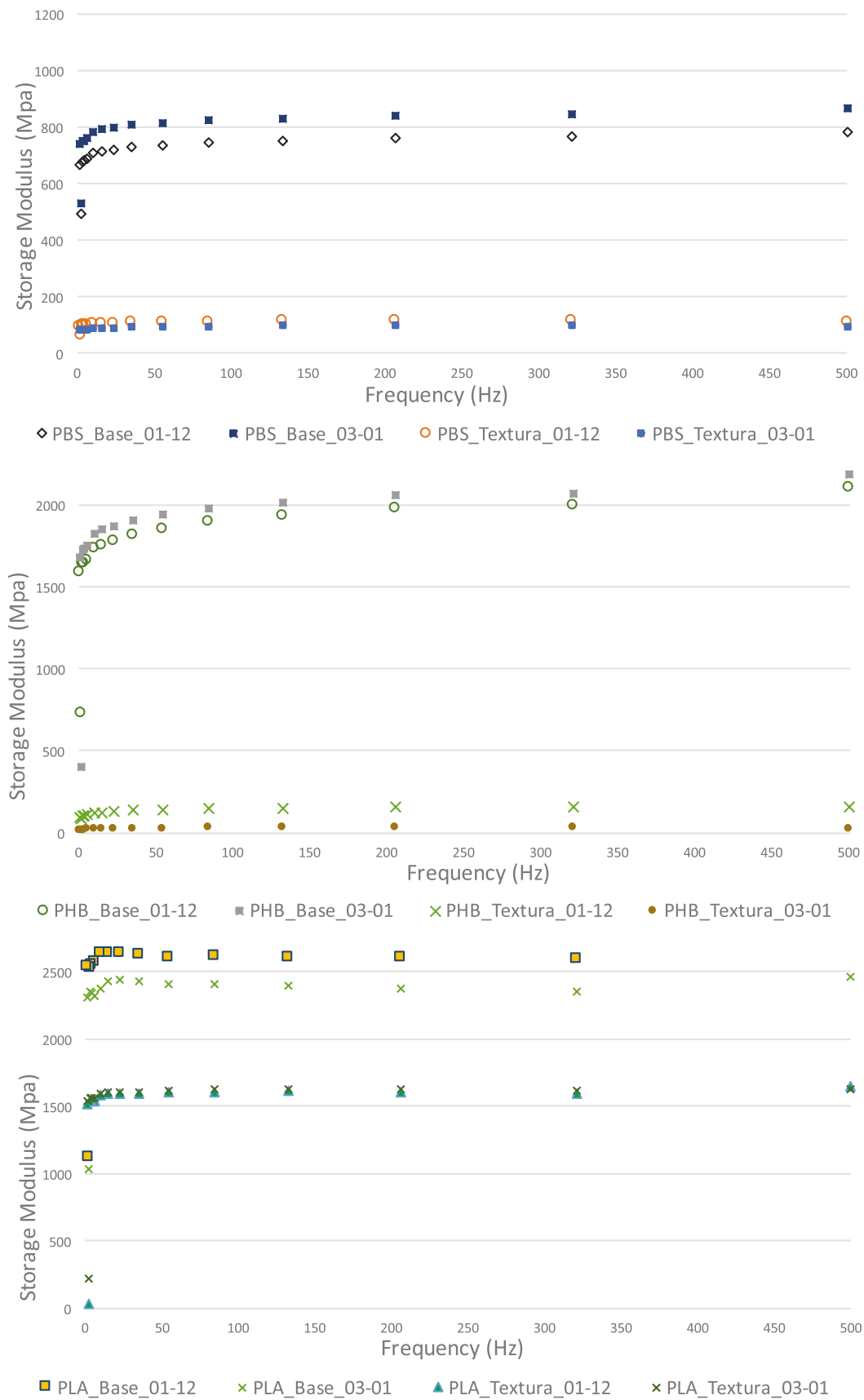
This fact is also supported by the progressive delamination of the micro/nanotextured Ormostamp<sup>®</sup> coating from the base PET film that took place as the number of injection rounds increased (see supplementary information). Once initial coating delamination took place, the subsequent increased adhesion of the injected polymer to the base film PET material caused the damage of the film and therefore its deformation upon part demoulding. As an indication of this problem, using a single coated film up to twelve samples of injected plastic were replicated for PBS and PLA (when the injection process had to be stopped due to substantial film-coating delamination) while up to fifty were replicated in the case of the PHB without critical film damage by delamination.

#### 3.2 Water contact angle

The contact angle measurements were taken for all the samples replicated with the same film (12 samples for PLA and PBS, 50 samples for PHB and 34 samples for PP) to account for potential variations in the hydrophobic character of the samples. The variations registered along the samples replicated were minimal as indicated in Table 2 (the values show the averaged values amongst the samples injected per each polymer, taking 5 measurements per part and zone).

**Table 3** COF results obtained for PHB, PLA, and PBS plastic parts after 4 days and after 30 days of the injection

Date tested	Polymer	Textured zone				Base material			
		0.5 N		1.5 N		0.5 N		1.5 N	
		Avg	SD	Avg	SD	Avg	SD	Avg	SD
01/12/2021	PHB	0.24	±0.032	0.19	±0.024	0.10	±0.015	0.10	±0.009
	PLA	0.15	±0.030	0.14	±0.029	0.07	±0.018	0.08	±0.011
	PBS	0.14	±0.040	0.12	±0.016	0.10	±0.010	0.09	±0.005
03/01/2021	PHB	0.26	±0.037	0.21	±0.029	0.12	±0.025	0.13	±0.019
	PLA	0.15	±0.021	0.15	±0.017	0.10	±0.028	0.10	±0.010
	PBS	0.15	±0.025	0.14	±0.022	0.06	±0.017	0.07	±0.008



**Fig. 5** Charts showing the storage modulus measurements obtained of both textured and non textured zones at various frequencies for PBI (above), PHB (centre) and PLA (below)

All the values show a change in wetting character of the polymers from their traditional low hydrophilic levels to slightly hydrophobic except for PP, which is already hydrophobic without the surface with micro/nano-texturing, but for which an increase in the contact angle was also measured. None of the increases in the contact angles obtained represent a dramatic change in wetting character, as the three-level hierarchical micro/nano textured were not specifically designed for that purpose. Nevertheless, the increases observed show an interesting switch in the wetting character of the biopolymers which is added to other potential surface functionalities achievable by hierarchical micro/nano textured surfaces.

### 3.3 Mechanical characterization

The values of the coefficients of friction (COF) taken for both textured and non textured zones four days and one month after the injection moulding experiments are shown in Table 3.

Figure 5 shows the evolution of storage modulus at various frequencies for both textured and non-textured surfaces for each of the biopolymers.

The values observed and their differences for textured and non-textured zones remain fairly constant along the time period tested. A slight difference of the COF between non-textured and textured zones can be observed for all the loads applied and the materials tested as expected.

Also, while a general slight increase of the COF values for both load levels along the time period tested is observed on all the textured zones of all the polymers, a small decrease is observed on values corresponding to the PBS base material. This decrease in the COF could be explained by the slight rise observed in the material's surface hardness along the defined testing time period, as appreciated in the measurements of storage modulus shown in Fig. 5-1 for the PBS non-textured surface.

In Fig. 5, lower values of storage modulus can be observed for the textured versus the non-textured zones for all the biopolymers tested as logically expected. Slight increases of the storage modulus between both dates tested are observed for the non-textured surfaces of PBS and PHB materials, while a more marked decrease is observed for the non-textured PLA. This could be explained by the known subtle degradation of PLA material mechanical properties degradation [26], potentially enhanced by the specific additives used by the manufacturer of the specific polymer grade used in the experiments. Additionally, very small (and therefore not conclusive) or negligible changes of the values observed on the textured zones of the different biopolymer materials are shown on the graphs.

## 4 Conclusions

A method for the fabrication of polymeric hierarchical inserts for isothermal plastic injection moulding using low-cost and recyclable materials has been developed.

A method to produce high accuracy, hierarchical micro-structured moulds at wafer scale, and its replication into plastic inlays is described. The method is scalable and affordable, as once the mould is fabricated, any of the intermediate replications can be used to define additional inlays.

The high degrees of replication filling (DR%) achieved on the samples injected show the feasibility of the method presented for the replication of hierarchical micro/nanotextured on the surfaces of common thermoplastic biopolymers using standard injection moulding conditions and polymeric flexible tooling. This is further underlined by the clear replication of the three hierarchical levels in most of the cases (shown on more than 80% of total structured surfaces on replicated samples in which no damage on film had occurred).

Amongst the biopolymers tested, PHB shows the most promising characteristics, as with the method applied it achieved the highest number of parts with a constant degree of replication and contact angle using a single film inlay.

The research presented here constitutes a promising low cost method using recyclable tooling and standard injection moulding conditions to achieve a sufficient contact angle increase on the surface of the biopolymers considered (hydrophilic on their original state) without substantial loss in surface mechanical properties, which might be of interest for applications such as packaging or the medical device industry. However, some improvements need to be made in order to enhance the film-coating durability and flatness during the injection moulding process to adequately increase the number of injected parts per film. Also, an adjustment of the cooling times and demoulding processes is required to obtain higher durability of the tooling.

**Supplementary Information** The online version contains supplementary material available at <https://doi.org/10.1007/s00170-022-10338-5>.

**Funding** Open Access funding provided thanks to the CRUE-CSIC agreement with Springer Nature. Carlos Sáez-Comet received funding from “Departament d’Economia i Coneixement de la Generalitat de Catalunya” in the frame of the “Doctorats Industrials” program. J.P. received financial support from T the Spanish Ministry of Economy and Competitiveness for the Project RTI2018-101827-B-I00 and the Generalitat de Catalunya for the grant 2017SGR373. Part of this work has been performed within the PLASTFUN project within the Industries of the Future community (IDF) RIS3CAT supported by the European Regional Development Fund (ERDF) as part of the operative frame FEDER of Catalonia 2014–2020 EC [COMRDI 16–1-0018], included in the 7th Framework Program and also within the Industrial Doctoral Program of Generalitat de Catalunya.

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written consent for participation in the work and publication of the results. The results are appropriately placed in the context of prior and existing research. All sources used are properly disclosed. All authors have been personally and actively involved in substantial work leading to the paper, and will take public responsibility for its content.

## Declarations

**Conflict of interest** The authors declare no competing interests.

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