Surface wear of TiN coated nickel tool during the injection moulding of polymer micro Fresnel lenses

Tosello, Guido; Hansen, Hans Nørgaard; Gasparin, Stefania; Albajez, José Antonio; Esmoris, José Ignacio

Published in:
C I R P Annals

Link to article, DOI:
10.1016/j.cirp.2012.03.016

Publication date:
2012

Citation (APA):
Tosello, G., Hansen, H. N., Gasparin, S., Albajez, J. A., & Esmoris, J. I. (2012). Surface wear of TiN coated nickel tool during the injection moulding of polymer micro Fresnel lenses. *C I R P Annals*, *61*(1), 535-538. https://doi.org/10.1016/j.cirp.2012.03.016

G. Tosello et al. (2012) Annals of CIRP, Vol. 61/1, 535-538, DOI 10.1016/j.cirp.2012.03.016

Surface wear of TiN coated nickel tool during the injection moulding of polymer micro Fresnel lenses

Guido Tosello (2)*[1], Hans Nørgaard Hansen (1)[1], Stefania Gasparin[1], José Antonio Albajez[2], José Ignacio Esmoris[3]

[1] *Department of Mechanical Engineering, Technical University of Denmark (DTU), Building 427S, Produktionstorvet, DK-2800, Kgs. Lyngby, Denmark*

[2] *Department of Design Engineering and Manufacturing, University of Zaragoza, C/María de Luna 3, E-50018, Zaragoza, Spain*

[3] *Department of Micro and Nanotechnology, TEKNIKER, Otaola 20, E-20600, Eibar, Spain*

*Corresponding author: *guto@mek.dtu.dk*

Abstract

Limited tool life of nickel mould inserts represents an issue for the mass-production of polymer optics with complex micro three-dimensional geometries by injection moulding. TiN coating was applied to a nickel insert for the injection moulding of polycarbonate micro Fresnel lenses. Surface wear was monitored at different intervals during production on different tool locations. 3D micro optical dimensional microscopy, surface replica technique and SEM-EDS were employed to characterize wear of the micro features. Results showed wear decreasing at higher distance from the gate. After 24500 moulding cycles the measured height reduction of 23 µm high ribs was on the order of 400 nm to 1000 nm.

Keywords: Micro tool, Wear, Moulding

1. Introduction

Injection moulding is an effective replication manufacturing technique for the mass production of high value optical components such as high precision lenses [1] and micro/nano optics [2,3]. Precision moulding of polymer micro structured surfaces with optical quality poses the highest challenges in terms of tooling and replication because of the combination of high geometrical complexity and high accuracy.

The crucial steps of the process chain for the manufacturing of polymer-based micro optics are: (a) the ultra-high accuracy micro machining of the mould cavity with nanometre surface finishing; (b) the achievement of an optimized injection moulding process to obtain high geometrical replication fidelity and optical surface finish of the plastic part; (c) the capability of maintaining a repeatable replication moulding process over a large number of cycles (i.e. mass production capability). This latter aspect is directly related to the moulding process repeatability itself (including the injection moulding machine, the process parameters, and the polymer material), and also to the ability of the mould cavity to maintain its geometrical characteristics for the whole production. This means that the tool wear should not compromise the integrity of the tool and consequently the quality of the moulded optical part (especially on the micro structured area).

Nickel tools allow the generation of polymer optics with complex micro three-dimensional (3D) geometries by injection moulding. However, limited tool life of nickel inserts represents an issue in preserving the original micro-geometry over a large number of moulding cycles (i.e. cycles number $>10^4$). Therefore, a titanium nitride (TiN) coating was applied to a micro structured nickel insert for polymer micro Fresnel lenses manufacture and tested by injection moulding for a large number of cycles in a production environment. The micro Fresnel lenses are designed for a miniaturized portable lighting device for digital imaging with a production volume forecast in the order of 10^5 specimens/year, therefore a relatively long tool life is of paramount importance. The use of TiN hard tribological coatings has been investigated in order to increase the wear resistance of substrates such as ceramic and hard metal (e.g. WC-Co) cutting tool inserts [4,5] as well as forming dies [6].

The aim of the present study was to experimentally assess the tool life and in particular the coating wear resistance during production. Recent studies have focused on the life of tools made of non-conventional materials such as aluminium and brass [7] for injection moulding, as well as photopolymer [8] for short production series in micro injection moulding applications. However, it appears that a lack of actual data for tool life during mass production still exists, especially for micro structured nickel tools. The main factors investigated in the present research were the evolution of the wear on the surface micro structures as a function of: (a) the effect of TiN coating; (b) the number of moulding cycles (surface wear was monitored at different intervals during the production until 24500 cycles); (c) the lenses distance from the injection gate.

2. Experimental

2.1 Tooling

The mould insert is made of nickel (Ni), obtained using an electroforming deposition process [9,10]. The primary master geometry used for deposition is made of aluminium and generated by ultra-high-precision diamond cutting technology and it is subsequently replicated by nickel electroforming. The result is a highly 3D micro Fresnel lens geometry (see Figure 1) with an average surface roughness Sa=3.3 nm measured by atomic force microscopy (AFM) (with an expanded measuring uncertainty of 1.3 nm, k=2 and confidence level of 95%, evaluated applying the GUM [11] and the method described in [12]) suitable for optical applications. Two nickel-plated plates, each containing six pairs of Fresnel lenses, were manufactured and cut by wire electrical discharge machining on a 30 mm x 80 mm rectangular shape in order to be mounted on a two-cavity flexible mould capable of accommodating differently structured mould inserts (see Figure 2, left). One of the plates was then TiN coated. By mounting both plates in the two-cavity flexible mould, it was possible to mould polymer Fresnel lenses from both the coated and the uncoated insert during the same moulding cycle and therefore with analogous moulding processing conditions (Figure 2, right).

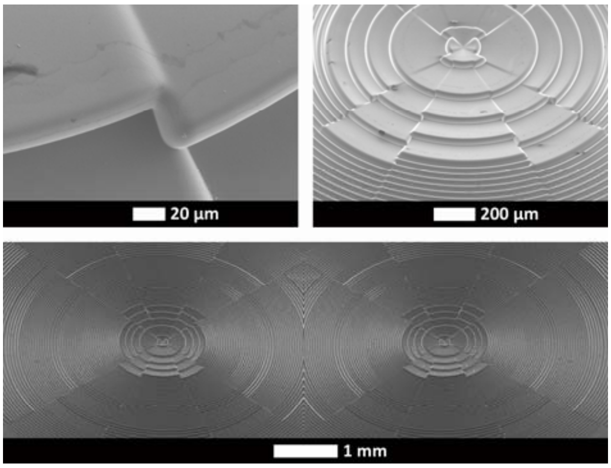

Figure 1. Micro Fresnel nickel tool surface geometries.

Figure 2. Two-cavity flexible mould with both coated and non-coated micro Fresnel nickel tool inserts mounted (left); two-parts moulded parts: the six pairs of micro Fresnel lenses are visible on the surface of both components (material = polycarbonate).

2.2. TiN coating

TiN coatings were prepared on the electroplated nickel substrate using a reactive pulsed magnetron sputtering. Magnetron sputtering is a widely used physical vapour deposition (PVD) technology employed to deposit thin films based on the generation of a magnetically enhanced glow discharge (i.e. the magnetron discharge). When a reactive gas such as nitrogen or oxygen is added to the discharge, it becomes possible to deposit the desired compound materials depending on the target material. For the present case, a 6” diameter target made of high purity titanium (99.95%) was employed for the deposition of the coating. A DC power supply (Advanced Energy, model No. MDX-1.5K, 1.5 KW) and an asymmetric bipolar-pulsed DC power supply (ENI, model No. RPG-50, 1.5 kW, 250 KHz, 1600 ns pulse width) were used to sputter the targets. Prior to deposition, the Ni substrate was cleaned in an ultrasonic stirrer using acetone, isopropyl alcohol and deionised water. The vacuum chamber was evacuated to a base pressure of 5.0×10^{-3} mTorr using a turbomolecular pump backed by a rotary pump. The substrates were subjected to an Ar+ ion bombardment at a substrate bias of −130 V and a pressure of 10 mTorr for 30 min to remove remaining impurities. Also, prior to the deposition, the targets were sputter cleaned for 5 min. The flow rates of argon and nitrogen were controlled separately by mass flow controllers. The coatings were deposited at a substrate temperature of 250 °C and at an Ar+N_2 gas pressure of 10 mTorr during 4 hours. The process was aimed at obtaining a final coating thickness of 2 µm. As a result of the TiN deposition, the surface roughness of the tool insert increased to 13.6 nm (with an expanded measuring uncertainty of 4.0 nm, k=2 and confidence level of 95% [11,12]). The newly obtained surface roughness is still suitable for optical application. The deposited TiN particles are also visible on the surface of the coated tool as compared with the uncoated Ni surface (see Figure 3). A tribology test and a nanoindentation hardness test were performed to characterize the TiN coated surface characteristics as compared with the uncoated Ni surface.

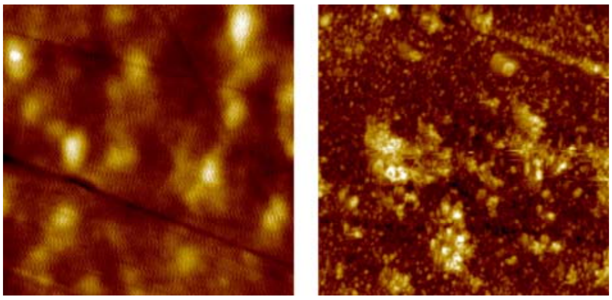

Figure 3. AFM scannings (15 µm × 15 µm) of the nickel surface (left, Sa=3.3±1.3 nm) and the TiN coated surface (right, Sa=13.6±4.0 nm).

The tribology test was performed to determine the ability of the coating to resist wear under linear-oscillation motion. For this purpose, a tribometer consisting of an oscillation drive, a test chamber with a loading device with a servomotor and a load cell, is used to simulate reciprocating sliding motion. This test configuration has been selected as the closest approximation to the shear condition encountered by the tool surface during the actual injection moulding process. Coefficients of friction (COF) are recorded in relation to time by an automated data acquisition system. The tribometer comprises a fixed bottom specimen support and a mobile, replaceable top specimen support. The upper and lower specimens are pressed against each other by an adjustable force and are oscillated tangentially, employing and adequate frequency/stroke combination. In the present test, a "ball-on-disc" configuration was employed. The test sphere oscillates at constant frequency and stroke amplitude and under constant load, against the coated test piece. The platform to which the test piece is attached is held at a constant temperature. The COF is determined during the test by measuring the deflection of an elastic arm by direct measurement of the change in torque. The measurement of the friction force is monitored continuously and the evolution of the mean friction coefficient is recorded. The applied test conditions were set as follows: applied load 10 N, oscillation frequency 10 Hz and 50 Hz, oscillation amplitude 1 mm, test temperature 25 °C, test duration 30 minutes. The material of the test ball was ultra-high molecular weight polyethylene (UHMWPE) with an average surface roughness of 1 µm. Results show that the TiN coated surface has a slightly higher coefficient of friction than the uncoated nickel. Wear of the polyethylene sphere is higher with the TiN coated disk at 50 Hz and adhesions on the disk surface occurred (see Table 1).

Table 1. Tribology tests results.

Surface	Frequency	COF±U (k=2, 95% conf.)	Wear on specimen surface
Ni	10 Hz	0.40±0.03	Negligible
TiN	10 Hz	0.53±0.02	Negligible
Ni	50 Hz	0.61±0.03	Negligible
TiN	50 Hz	0.68±0.02	Adhesions

Nanoindentation has established itself in the last decade as the fundamental quasi-non-destructive method for the evaluation of the mechanical response of small material volumes and thin films to applied loading [13,14]. As such, nanoindentation tests were performed on both uncoated Ni and TiN coated specimen to evaluate the increase of hardness of the insert surface as consequence of the coating deposition process. For this purpose, a Fischerscope H100 micro hardness tester system with a Vickers nanoindenter was employed. The employed test conditions included an increasing load (0.4-10 mN), applied in a series of 25 consecutive steps, up to a maximum indentation depth of 0.260 µm and 0.135 µm (equivalent to approximately 7 % of the coating thickness) for the Ni and TiN coated specimens respectively. Residual indentation depth of 0.238 µm and of 0.073 µm were obtained for the Ni and TiN coated specimens respectively with a repeatability of ±0.003 µm. Indentation hardness of 3.00±0.15 GPa for the Ni insert and of 12.77±0.18 GPa for the TiN insert were obtained.

2.3. Injection moulding

Polymer micro structured optical Fresnel lenses were injection moulded using a commercially available optical grade high-flow polycarbonate (Makrolon 2405 by Bayer MaterialScience). Injection mouldings were executed on a conventional injection moulding machine with a reciprocating screw of diameter of 35 mm and a clamping force of 60 kN. The injection moulding was set in order to provide similar conditions to those encountered during actual processing in order to comply with industrial requirements such as cycle time, optical functionality (i.e. surface replication), and tool wear conditions as experienced in production. For these reasons, a melt temperature of 295°C was selected close the maximum recommended from the material supplier in order to avoid polymer overheating and subsequent material degradation, which could compromise the optical performance of the lens, and to optimize polymer surface replication [15]. A mould temperature of 90°C was set following the recommendations from the material supplier, in order to allow successful demoulding of the part from the cavity, to perform the injection process within a suitable cycle time (i.e. short cooling time), and maximize surface replication [15]. An actual injection speed of 200 mm/s was used, taking into account the machine capability. A high injection speed allows improving surface replication because reduces cavity injection time avoiding polymer premature freezing. A maximum injection pressure of 900 bar was reached. A total cycle time of 15 s was obtained including packing, cooling, and demoulding phases.

2.4. Micro dimensional geometrical metrology

The dimensions of the tool inserts micro structures were investigated at different production stages to evaluate the tool wear progress. However, the characterization of highly reflective surfaces with nanometer surface finish is a challenging task to be performed. On one hand, tactile instruments could damage the surface itself, and on the other hand, measurements carried out with optical instruments could be hampered by the high reflectivity of the surface limiting their accuracy. Also, the inserts have to be dismounted at each quality control step of the quality control process to be positioned on the measuring instrument, making the whole procedure lengthy and cumbersome. For these reasons, an indirect surface metrology method based on surface replication was developed and applied directly on the tool using a soft replica material (polydimethylsiloxane, PDMS) curing at room temperature [15,16]. The polymer casting replication was performed using a compound supplied in cartridges containing both the polymer (PDMS) and the curing agent, which are automatically mixed in a disposable static-mixing nozzle during the application to the surface. A replication mould device that could be easily mounted and removed from the mould was designed and manufactured to solve the issue of disassembling the inserts from the injection moulding tool (Figure 4). Both mould inserts were cleaned using acetone prior the soft replication step to avoid that polymer residual could affect the replication fidelity of the surface.

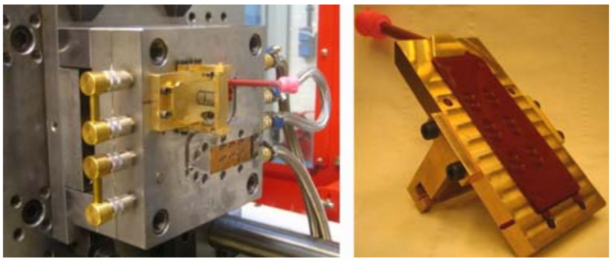

Figure 4. Replication mould device mounted on the injection moulding tool (left); replication mould device disassembled from the injection tool and containing the soft replica of the insert (right).

Once the replicas of the tool inserts surface were produced, they were measured with a focus variation three-dimensional optical profilometer which combines the small focus depth typical of an optical system, a controlled vertical scanning axis and 3D software rendering of planar 2D calibrated images. The traceability of measurements with such instrument on soft replicas of micro dimensional reference standard objects was investigated and an expanded combined uncertainty of 0.4 µm has been obtained [16]. Further, reproducibility studies on the tool replicas were conducted to ensure the reliability of the measurements over different periods of time. As a result, measurements on replicated micro structures with a height of 23 µm (see Figure 5) and repeated within few minutes, 3 hours and 24 hours for 14 days could be reproduced within a range of 0.1 µm, 0.3 µm, and 0.4 µm respectively.

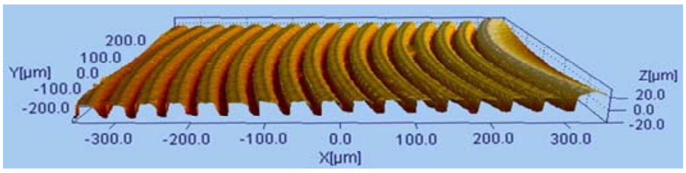

Figure 5. Result of a three-dimensional optical scanning of the Fresnel lens tool soft polymer replica on the area with 23 µm high micro ribs.

3. Tool wear assessment results

The tool wear was investigated by means of dimensional measurements (with the technique presented in §2.4), scanning electron microscopy and energy dispersive spectroscopy (SEM-EDS). A total number of 24500 injection moulding cycles were run. Dimensional measurement were carried out with the replica technique each 1000-2000 cycles with the tool inserts mounted on the mould during production. On the contrary, the SEM-EDS techniques needed the inserts to be dismounted in order to be placed inside the scanning electron microscope.

3.1. Dimensional measurements

Optical measurements were carried out on soft replicas of both uncoated Ni and TiN coated tool inserts, on the nearest lens to the gate and on the furthest lens from the gate. The average height of 10 ribs as depicted in Figure 5 was chosen as the factor to monitor the tool wear progress. To calculate the rib height, a height distribution frequency analysis was performed; such analysis allowed excluding the influence of ribs sidewalls, including only the bottom and the plateau regions. Results showed a reduction of the average ribs height in the range between 0.7 and 1.1 µm for the uncoated lenses (both near and far from the gate) and the TiN coated lenses near the gate, whereas the TiN coated lenses far from the gate exhibit a height decrease of about 0.4 µm (see Figure 6).

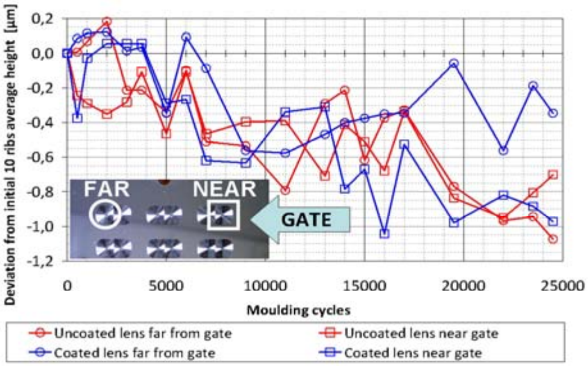

Figure 6. Deviation of the average 10 ribs height from the initial value before production for coated and uncoated lenses as a function of distance from the gate. Nominal average height of the ribs is 23 µm.

3.2. SEM-EDS inspection

An SEM inspection was carried out on the coated and uncoated mould after 0, 5000, 7000, 17000 and 24500 cycles in order to have a direct visual characterization of the wear progress. The investigation showed that until 17000 cycles no wear could be observed on the tool surface. However, after this point, at 24500 cycles, craters distributed on several zones of the TiN coated surface appeared as result of the structural deterioration of the coating. In particular, a delamination failure mode of the coated lenses near the gate can be seen in Figure 7 (b,c); on the contrary, surface integrity of the coated lens far from the gate and of Ni uncoated tool is shown in Figure 7 (a,d respectively).

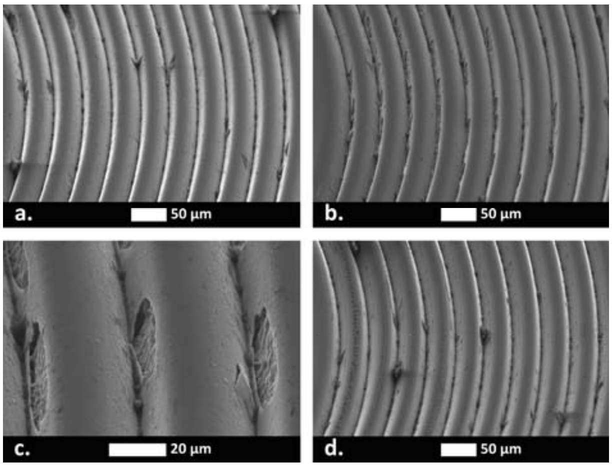

Figure 7. Craters on the TiN coating after 24500 moulding cycles not present on lens far from the gate (a), and occurring on lens near the gate (b)(c); corresponding area near the gate on the Ni uncoated tool without surface failure (d).

To complete the investigation with the scanning electron microscope, an EDS analysis of the material composition of both tool inserts was carried out (see Table 2). A 7.5% decrease of the Ti content on a 2 µm thick section of the surface was observed after 24500 cycles, proving the tool wear progress. This result was verified by performing the EDS analysis inside the craters themselves. The inspection showed a Ti content value of 14.3% and 85.7% of Ni. The substrate at the bottom of the craters is still part of the TiN coating, but a structural failure occurred.

Table 2. EDS analysis results.

No. cycles	TiN coated tool		Ni uncoated
	Ti [%]	Ni [%]	Ni [%]
0	99.8	0.2	100
5000	99.8	0.2	100
7000	99.9	0.1	100
17000	93.7	6.3	100
24500	92.5	7.5	100

4. Conclusion

A study on tool wear of TiN coated Ni injection moulding inserts for polymer optics application was conducted. A quantitative study based on a soft replica technique and 3D optical metrology combined with an SEM-EDS investigation was performed.

The tool wear progress was monitored and three different wear mechanisms were revealed: (a) a coating structural failure occurring at 24500 moulding cycles with craters appearing on the tool surface; (b) a progressive feature height reduction observed throughout the production; (c) a continuous decrease of Ti in the composition of the initial surface layer on the coated tool.

The surface wear of micro structured TiN coated nickel tool reached a critical level at 24500 moulding cycles. Despite the high hardness of the coating (as shown by the indentation hardness test), the coating showed a structural failure under the cyclic thermo-mechanical load typical of the injection moulding process, where high temperatures and pressures are repeatedly reached during the filling of the cavity and decrease during the packing and cooling phases. The coating wear was also promoted by the higher coefficient of friction as proved by the tribological test.

Finally, the effect of the lens position relatively to the injection gate was highlighted: it had the effect of decreasing the tool wear of the TiN coated insert (i.e. lower deviation from initial dimension and no craters on the surface) on lens far from the gate. This relates with the lower pressure at the end of the cavity due to the pressure drop experienced by the polymer far from the injection location. Indeed, even though measurements on the tool micro features still showed a height decrease during the production, a slight better performance of the TiN coating far from the gate (where the thermo-mechanical conditions are less severe) was observed.

Acknowledgements

This paper reports work undertaken in the context of the project ‘‘COTECH-Converging technologies for micro systems manufacturing’’. COTECH (http://www.fp7-cotech.eu/) is a Large Scale Collaborative Project supported by the European Commission in the 7th Framework Programme (CP-IP 214491-2). The support from the “Departamento de Ciencia, Tecnología y Universidad del Gobierno de Aragón” (Spain) which sponsored the research stay of Dr. José A. Albajez at the Technical University of Denmark (DTU) is gratefully appreciated. B.Sc. Anders Riel and B.Sc. Sture G. Bachmann (both DTU) are acknowledged for their contribution in connection with the moulding experiments and the design of the replication mould device.

References

[1] Michaeli W, Heßner S, Klaiber F, Forster J (2007) Geometrical Accuracy and Optical Performance of Injection Moulded and Injection-compression Moulded Plastic Parts, CIRP Annals - Manufacturing Technology, 56(1):545-548.
[2] Kang S (2004) Replication Technology for Micro/Nano Optical Components, Japanese Journal of Applied Physics, 43/8B: 5706-5716.
[3] Yoon S-H, Lee J, Park K, Mead JL, Matsui S, Barry CMF (2006) Critical factors for nanoscale injection molding, Smart Medical and Biomedical Sensor Technology IV, Proc. of SPIE Vol. 6380, 63800K1-12, 12pp.
[4] Grzesik W, Małecka J (2011) Documentation of tool wear progress in the machining of nodular ductile iron with silicon nitride-based ceramic tools, CIRP Annals - Manufacturing Technology, 60(1):121-124.
[5] Settineri L, Levi R (2005) Surface Properties and Performance of Multilayer Coated Tools in Turning Inconel, CIRP Annals - Manufacturing Technology, 54(1):515-518.
[6] Osakada K, Matsumoto R (2000) Fundamental Study of Dry Metal Forming with Coated Tools, CIRP Annals - Manufacturing Technology, 49(1):223–226.
[7] Hernandez P, Diaz M, Covelo E, Ares E (2011) Evaluation of the Wear During the Injection Moulding Process Using Optical Profilometry, 4th Manufacturing Engineering Society International Conf., Cadiz (Spain), 21-23 September 2011, 8pp.
[8] Griffiths CA, Dimov SS, Hirshy H, Scholz S, Fischer S, Spitzbart M (2011) Prototype tooling for producing small series of polymer microparts, Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture, 225(12): 2189-2205.
[9] Tang PT (2008) Utilising Electrochemical Deposition for Micro Manufacturing, Proceedings of the 4th International Conference on Multi-Material Micro Manufacture (4M), Cardiff (United Kingdom), 9-11 September 2008, 18-27.
[10] McGeough JA, Leu MC, Rajurkar KP, De Silva AKM, Liu Q, (2001) Electroforming Process and Application to Micro/Macro Manufacturing, CIRP Annals - Manufacturing Technology, 50/2:499-515.
[11] Joint Committee for Guides in Metrology (JCGM). (2008) JCGM 100:2008, Guide to the Expression of Uncertainty in Measurement (GUM), pp. i–viii, 1–132.
[12] Tosello G, Hansen HN, Marinello F, Gasparin S (2010) Replication and dimensional quality control of industrial nanoscale surfaces using calibrated AFM measurements and SEM image processing, CIRP Annals - Manufacturing Technology, 59(1):563-568.
[13] Lucca DA, Herrmann K, Klopfstein MJ, Nanoindentation: Measuring methods and applications, CIRP Annals - Manufacturing Technology, 59(2):803-819.
[14] Bruzzone AAG, Costa HL, Lonardo PM, Lucca DA (2008) Advances in engineered surfaces for functional performance, CIRP Annals - Manufacturing Technology, 57(1):750-769.
[15] Hansen HN, Hocken R, Tosello G (2011) Replication of micro/nano geometries, CIRP Annals - Manufacturing Technology, 60(2):695-714.
[16] Gasparin S, Hansen HN, Tosello G (2011) Traceable surface characterization using replica moulding technology, 13th International Conference on Metrology and Properties of Engineering Surfaces, National Physical Laboratory (NPL), Teddington (United Kingdom), 12-15 April 2011, 310-315.