Quality assessment of anti-sticking layers for T-NIL

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For defect-free separation of the stamp and the imprinted polymeric layer in a thermal nanimprint (T-NIL) process, F-saturated trichlorosilanes are employed typically, as e.g. 'F6' (CF_3-$(CF_2)_5$-$(CH_2)_2$-Si-Cl_3). These materials are capable of building up internally cross-linked monolayers that are chemically bonded to a hydrogenated Si surface, able to withstand imprint temperatures of up to 200°C without degradation[1]. Preparation can be conducted from the liquid phase or from the gas phase, the gas phase process claiming to be advantageous in case of fine, high aspect ratio stamp patterns[2]. For the latter two alternatives exist, (i) thermal evaporation of the liquid (boiling point 195°C) at atmospheric pressure or (ii) vacuum evaporation (vapor pressure 30 mTorr) at room temperature. Humidity control is a vital issue, as excess humidity favors gas phase polymerization and thus particulate contamination, wheras too low humidity hinders continuous layer formation. Controlled humidity is reported to improve the layer quality[3].

Though the main purpose of the F6 layer is sticking prevention, replication of optical components may require consideration of further aspects, like a specular surface. Our experiments indicate that this requirement may oppose good sticking prevention. As replication of optical structures via T-NIL requires both, good anti-sticking behaviour and good optical quality as well, the vacuum deposition process was investigated with respect to optimum conditions for this purpose. In addition to imprint experiments, providing qualitative information on separability, the quality of the anti-sticking layers was evaluated via contact angle measurement (see Fig. 1), but also via optical inspection of the surfaces. Normal reflection in the microscope reveals surface irregularities down to the micron range only, like small particles or droplets (see Fig. 2), whereas simple visual inspection under grazing illumination allows identification of even smaller contaminations otherwise requiring AFM techniques[4].

We have applied all four methods to assess the quality of vacuum deposited anti-sticking layers in order to identify an optimum preparation window for the deposition process in terms of F6 amount and deposition time. There are indications, that best anti-sticking performance occurs in a regime, where the surface quality is far beyond the requirements of optical components. Optical quality assessment requires experiments with specular surfaces as e.g. polished Si wafers, only highly contaminated surfaces would be detectable with a typical surface patterned stamp.

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1 R. Maboudian et al, Sensors and Actuators **82**, 219 (2000)
2 M. Beck et al, Microelectronic Engineering **61-62**, 441 (2002)
3 H. Schift et al, Nanotechnology 16, 171 (2005)
4 G.-Y. Jung et al, Langmuir **21**, 1158 (2005)

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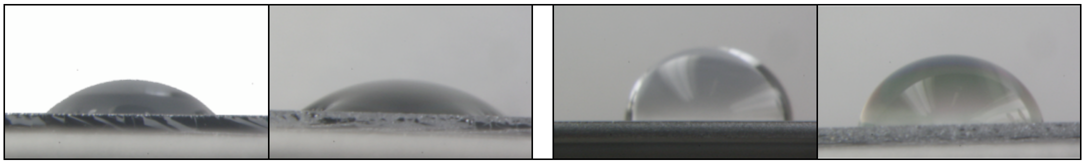

a1) a2) b1) b2)

Fig. 1: Characterisation by contact angle measurement (water on substrate).
Left: untreated Si: a1) plane surface, a2) patterned surface
Right: surface with anti-sticking layer: b1) plane, b2) patterned surface
On the patterned surface (1 μm structures) the contact angles are reduced.

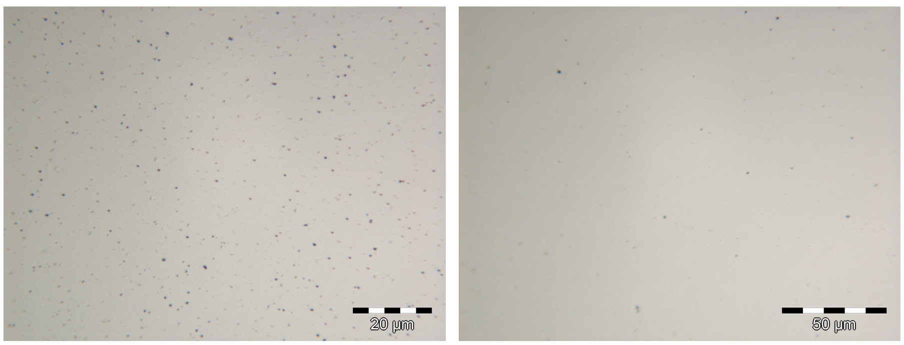

Fig. 2: Example for optical quality assessment of anti-sticking layers under the microscope.

Left: Numerous, large particles/droplets on the surface after deposition. They cannot be removed efficiently by a subsequent washing in solvent.

Right: Only few small particles are detectable after deposition, due to a reduced amount of F6 supplied for the process. The quality is still not sufficient for optical applications.

At sufficient optical quality, which is obtained at even lower amounts of F6, no more contamination is visible in the microscope.

10’	**97°**	**98°**	**106°**	**107°**	
20’	**94°**	**99°**	**101°**	**108°**	**106°**
30’				**109°**	**103°**
40’				**109°**	**104°**
	10µl	**15µl**	**20µl**	**25µl**	**30µl**

10’	**0**	**0**	**0**	**S**	
20’	**0**	**0**	**0**	**S**	**M**
30’				**S**	**M**
40’				**M**	**M**
	10µl	**15µl**	**20µl**	**25µl**	**30µl**

Fig. 3: Characterisation of anti-sticking layer quality by contact angle measurement (*left*) and optical inspection (*right*)

(O = no contamination under grazing incidence (best optical quality); S = faint particles visible under grazing incidence, M = first contamination visible in the microscope (low optical quality, Fig. 2b).

For the antisticking layers with the lowest optical quality the separation of stamp and sample after imprint is best.

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