# Surface replication of sub-µm structures
# Analysis, optimization and quality assurance

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EUSPEN workshop
**Session 4: Metrology for Micro/Nano Manufacturing**
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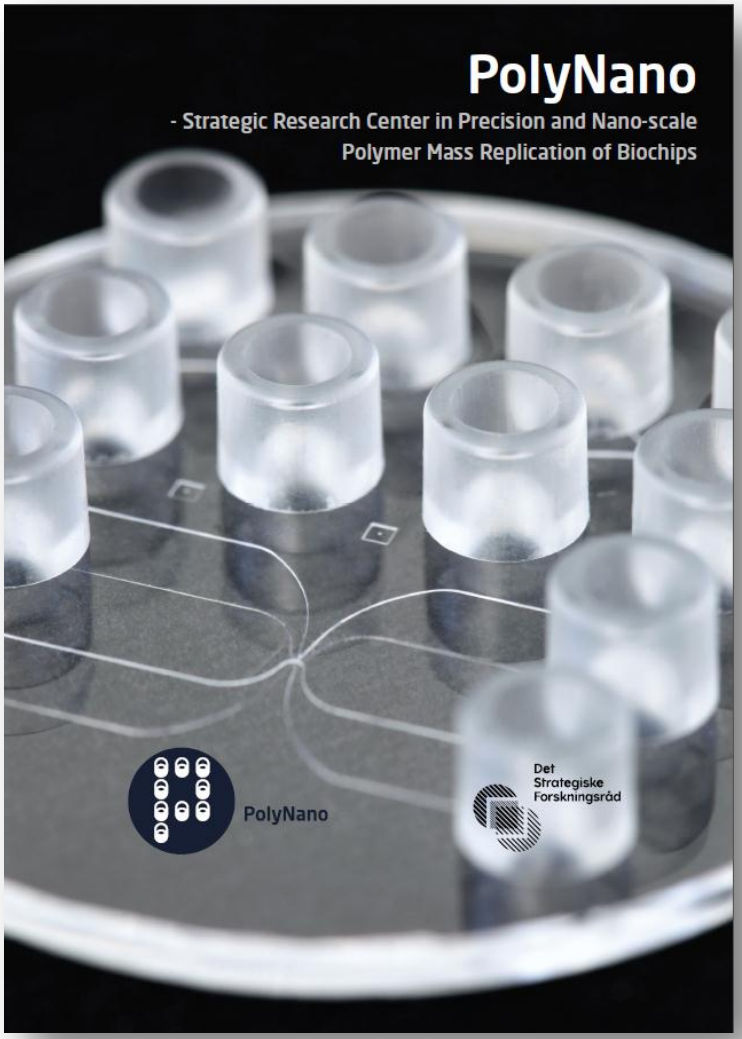
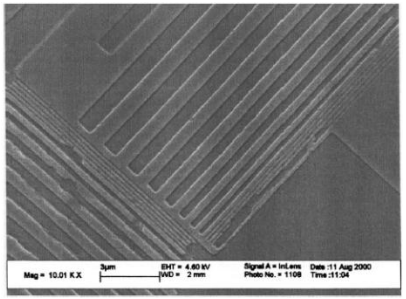
# Outline

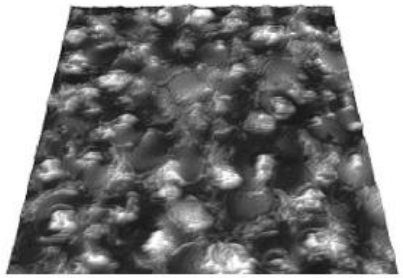
- Introduction
  - Quality control at nano scale
  - Aim of the study
- Moulds fabrication
  - Fabrication of deterministic nano structures
  - Fabrication of  distributed semi-spheres pattern
- Experimental injection moulding
  - Process analysis and optimization
- Dimensional measurements
  - Measurements results
  - Uncertainty assessment
  - Tolerance verification
- Conclusion, outlook

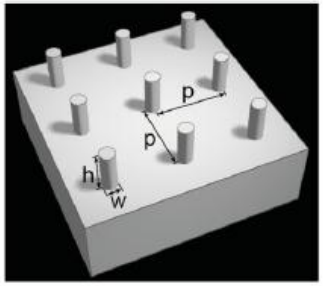


# Replication of sub-micro surface features

- Dimensional approaches to quality control of polymer structured surfaces by AFM measurements
- Polymer moulding capable of replicating sub-micro features
  - *Kim Y, Optical Engineering, 2002*
  - *Schift H, Microelectronic Eng., 2000*
  - *Heidari B, J. of Vacuum Sc. and Tech., 2000*
  - *Mönkkönen K, Polym. Eng. Sci., 2002*
  - *Theilade UA, Int J. Adv. Manuf. Technol., 2006*
  - *Matschuk M, J. Micromech. Microeng., 2013*
- **Typical replicated dimensions**
  - Width = 50-100 nm
  - Pitch = 100-500 nm
  - Height = 50-100 nm
- **PROCESS CALIBRATION**
- **METROLOGICAL APPROACH NEEDED**

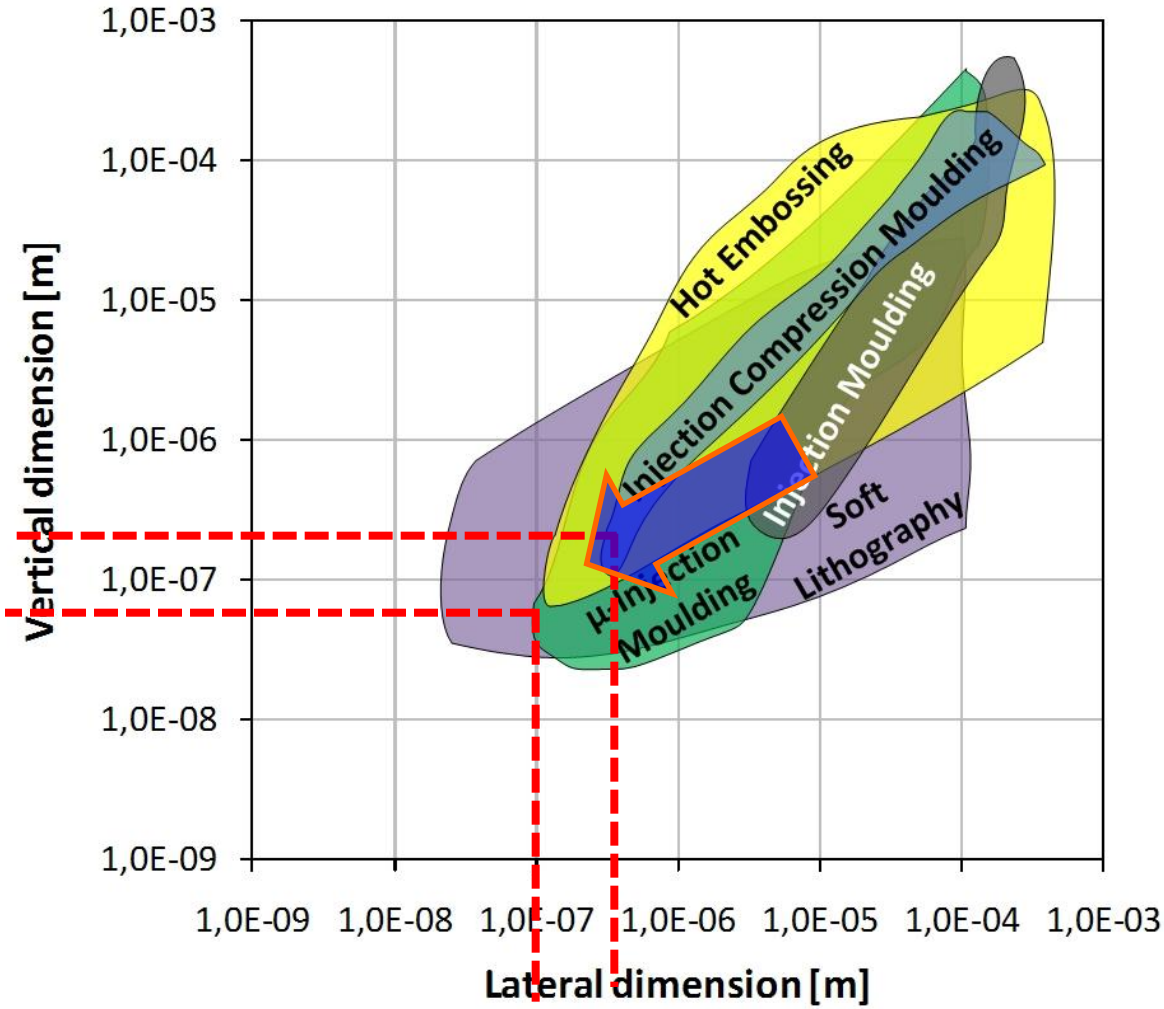






# Replication of sub-micro surface features

- State-of-the-art research conducted for replication of micro/nano geometries
- Injection and micro injection moulding, compression moulding, nano imprint technologies




*[Hansen, Hocken, Tosello, CIRP Annals 2011]*

# Mould fabrication
# Deterministic test structures

## DEEMO process

Dry Etching Electroplating and Molding

Oxidation with 100nm thick silicon oxide

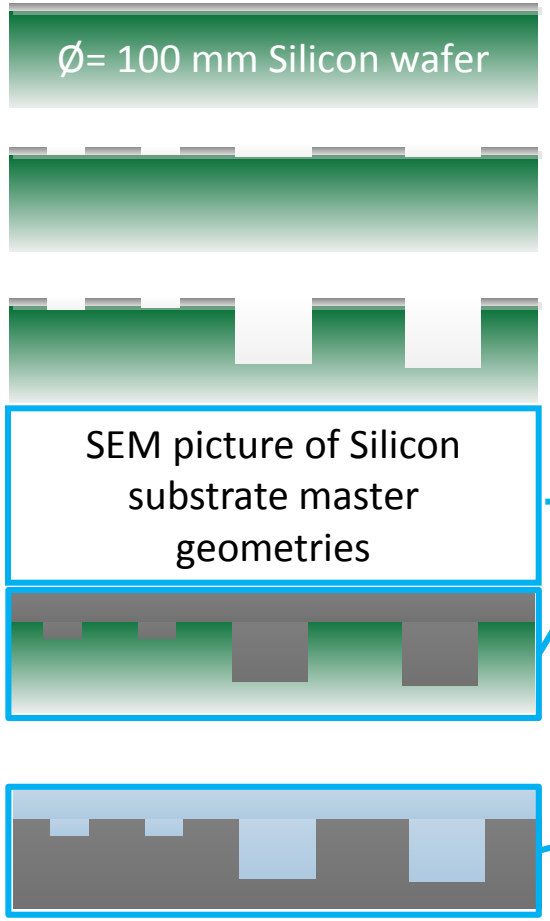
Oxide etching of shallow and micro channels

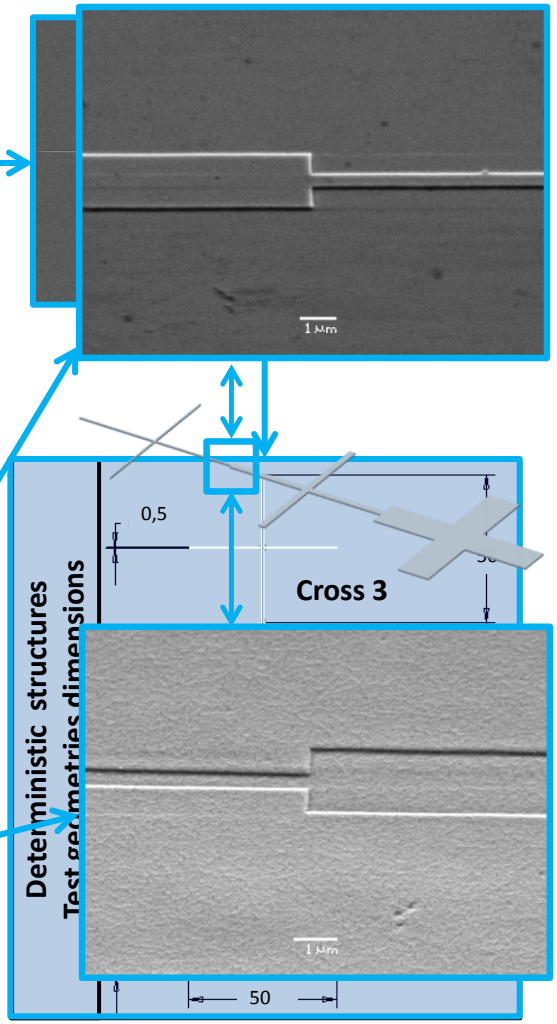
Dry Reactive Ion Etching (DRIE)of main channels

Reactive Ion etching (RIE) of the main channels

PVD → 10nm ti 100nm Au
Electroplating → 400µm nickel

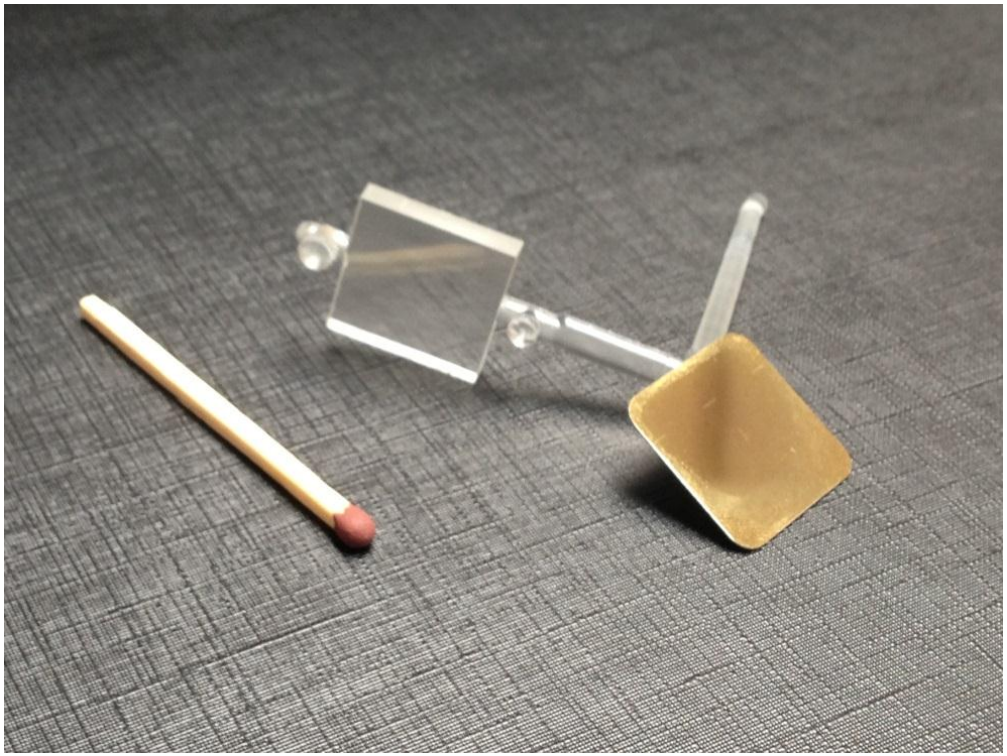
Injection moulding of COC Topas 6013 samples



# Experimental injection molding

Deterministic structures



Process parameters

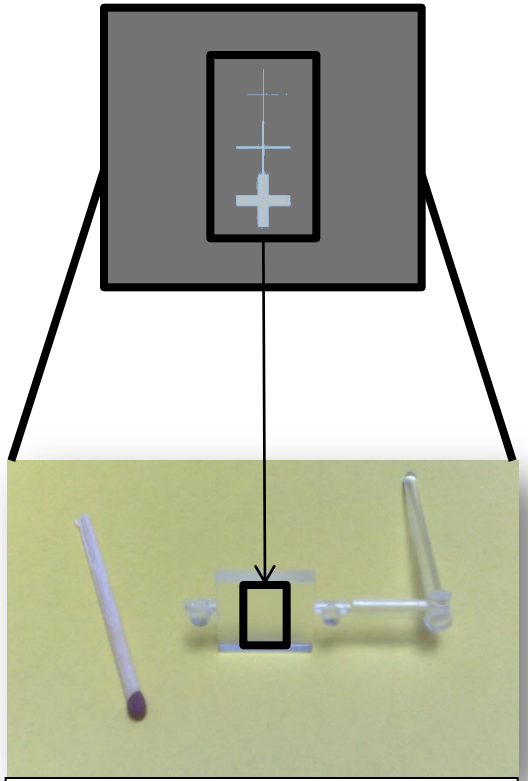
Mould temp. 130 ° C – Packing pressure 600 bar- Packing time 4,5 s

Melt temp. at 250° C and injection speed at 150 mm/s constant during experiments

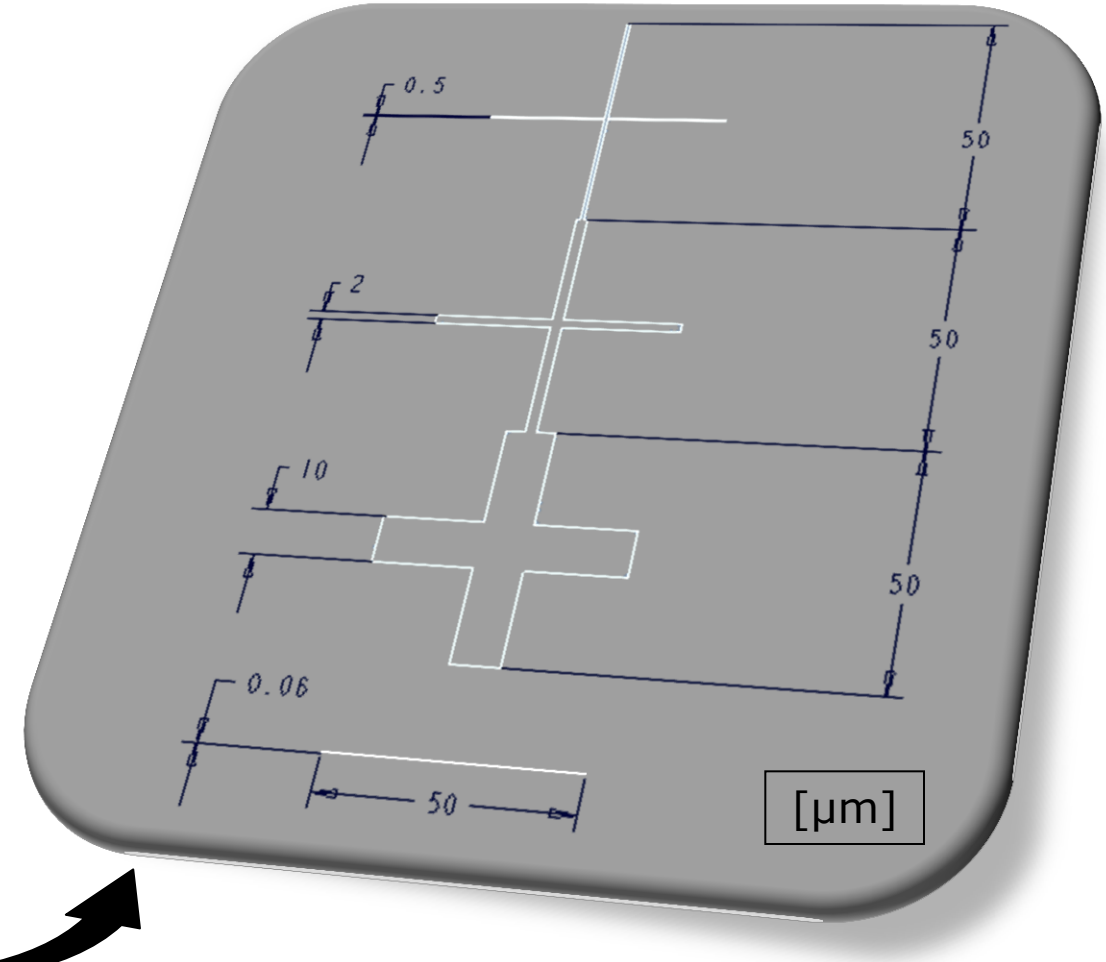
# Test geometries

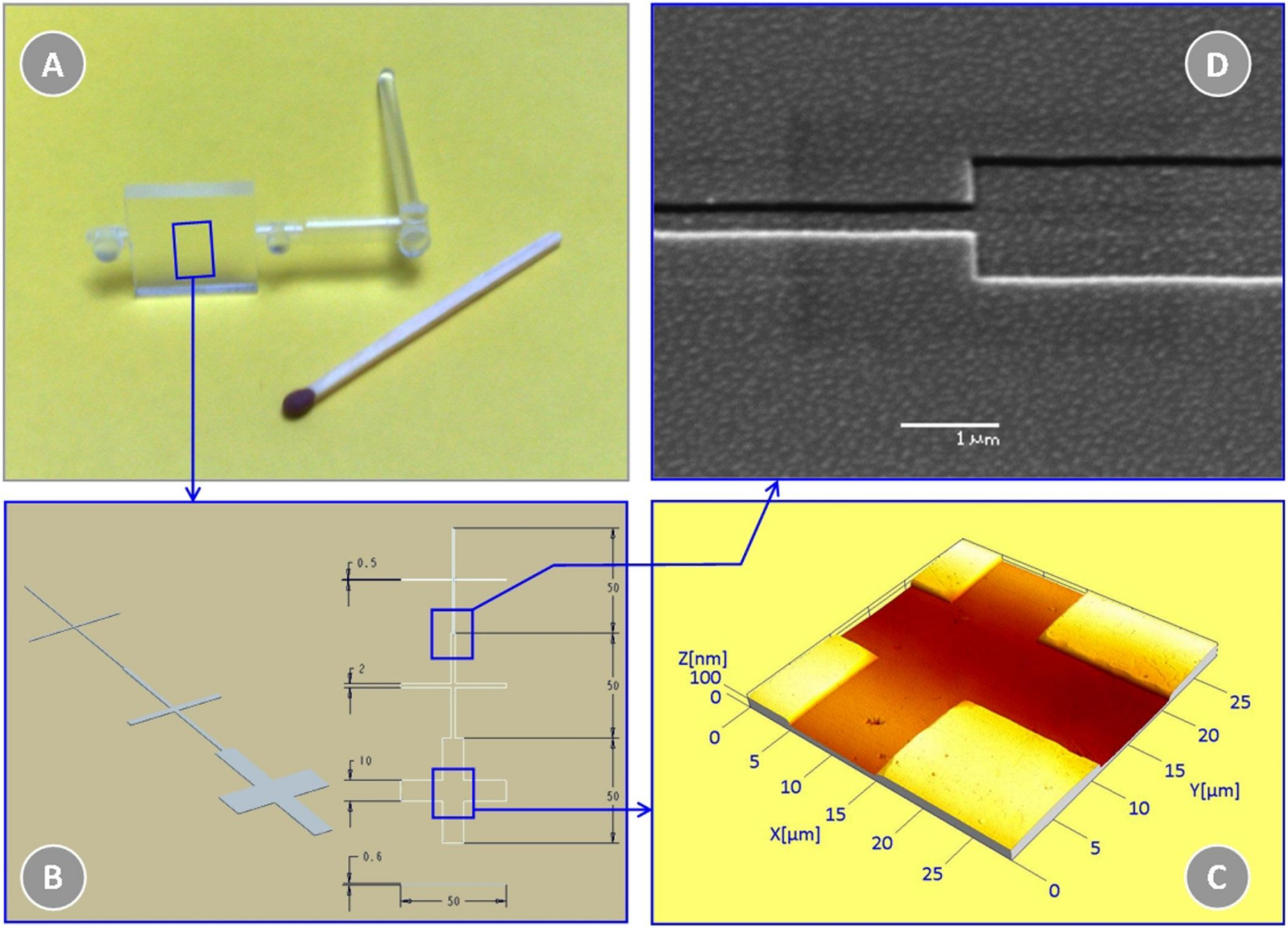
22x22 mm$^2$

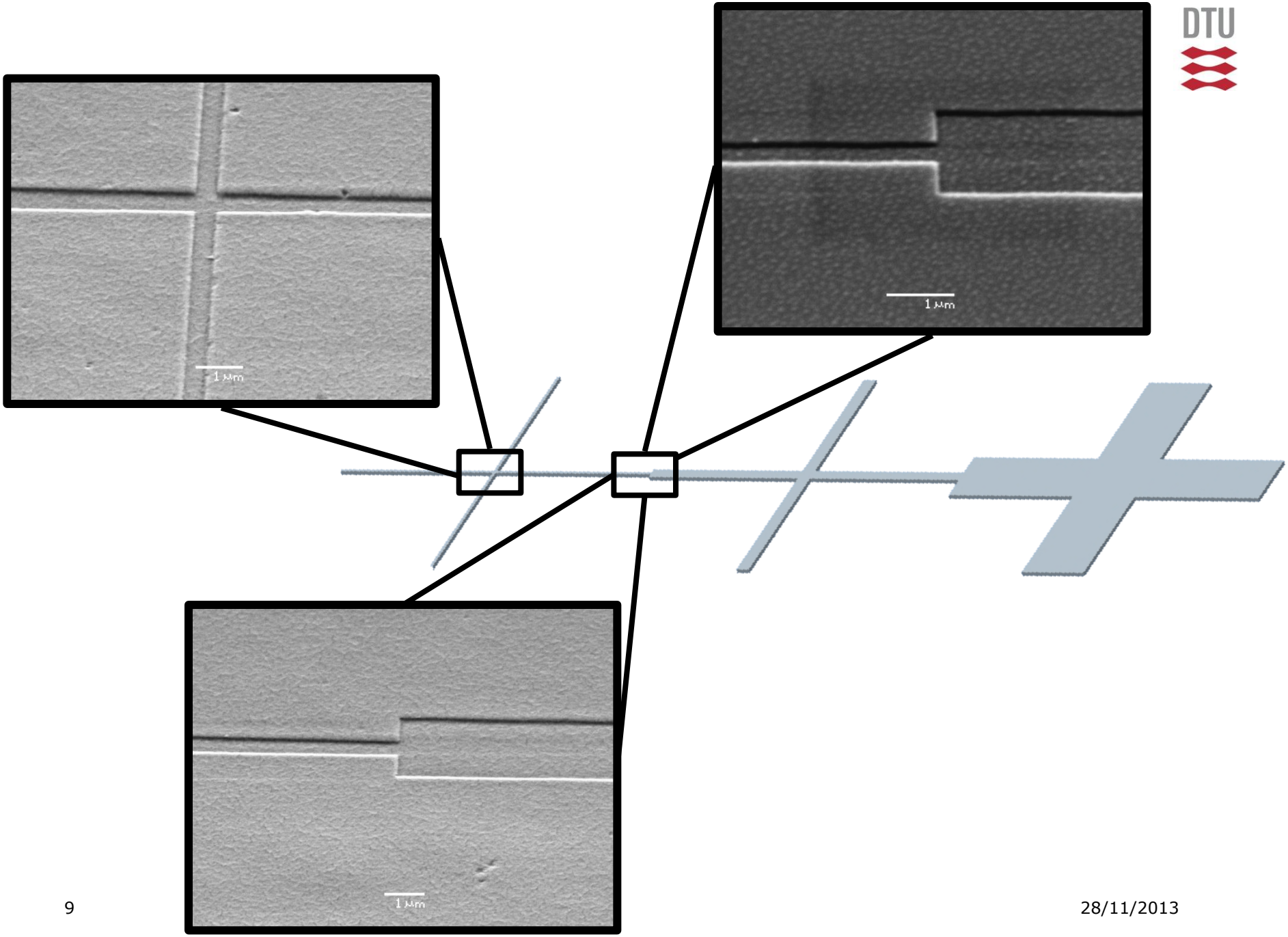
Nickel insert



Injection molded part



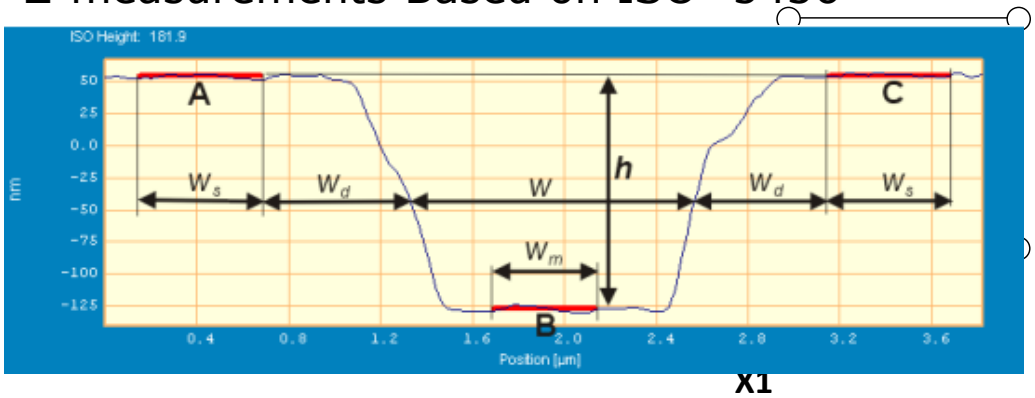

A
B
C
D
0.5
2
10
0.6
50
50
50
50
1 µm
Z[nm]
100
0
0
5
10
15
20
25
X[µm]
0
5
10
15
20
25
Y[µm]


1 µm
1 µm
1 µm

# Process optimization

## Two-level factorial design

Z-measurements Based on ISO -5436

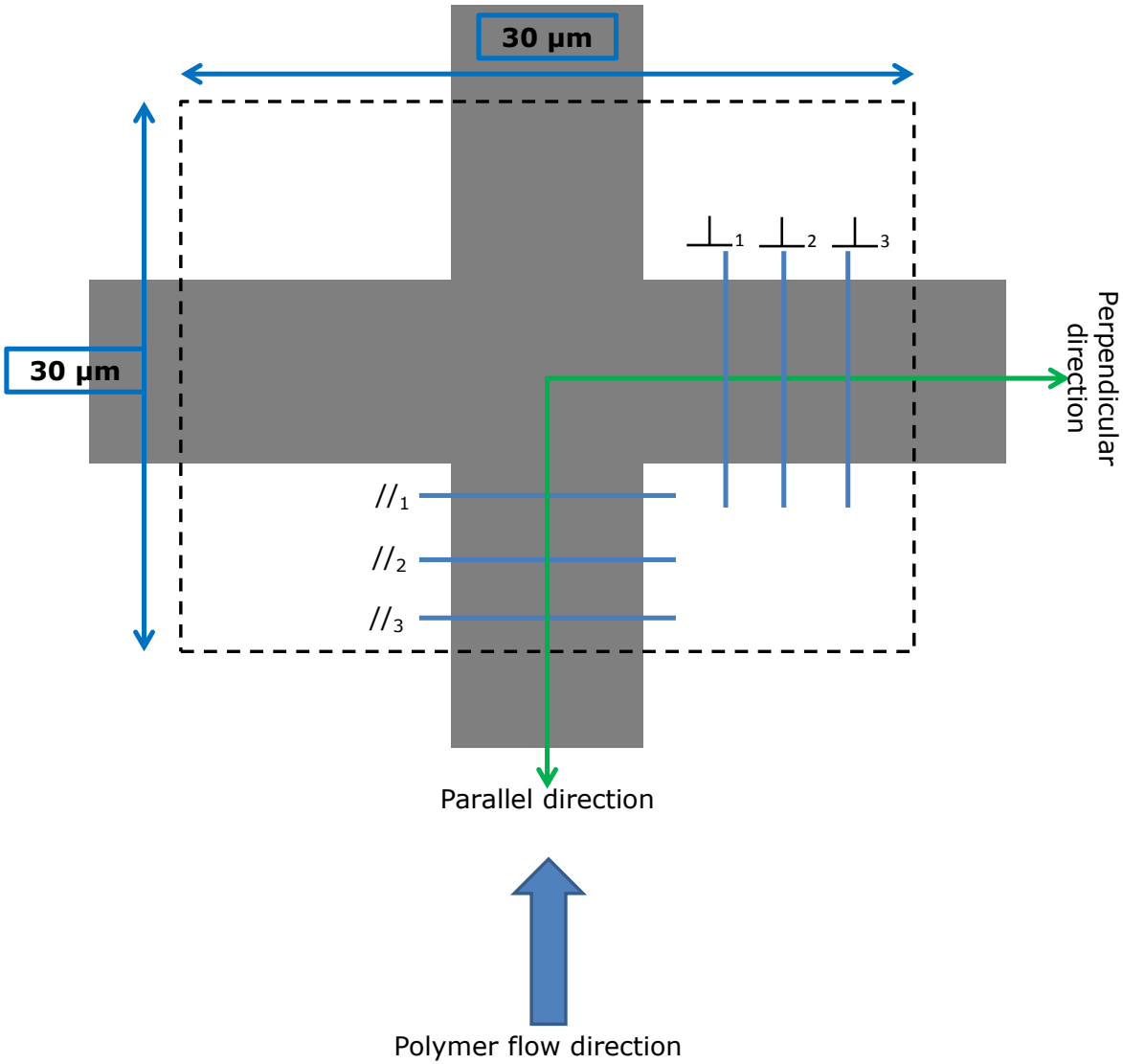



X1

| | Source of variation | Level (-) | Level (+) |
|---|---|---|---|
| **X1** | Mold temperature | 120° C | 130° C |
| **X2** | Packing pressure P(pack) | 400 bar | 600 bar |
| **X3** | Packing time t(pack) | 3 s | 4.5 s |

Melt temp. at 250° C and injection speed at 150 mm/s constant during experiments

# Measuring strategy



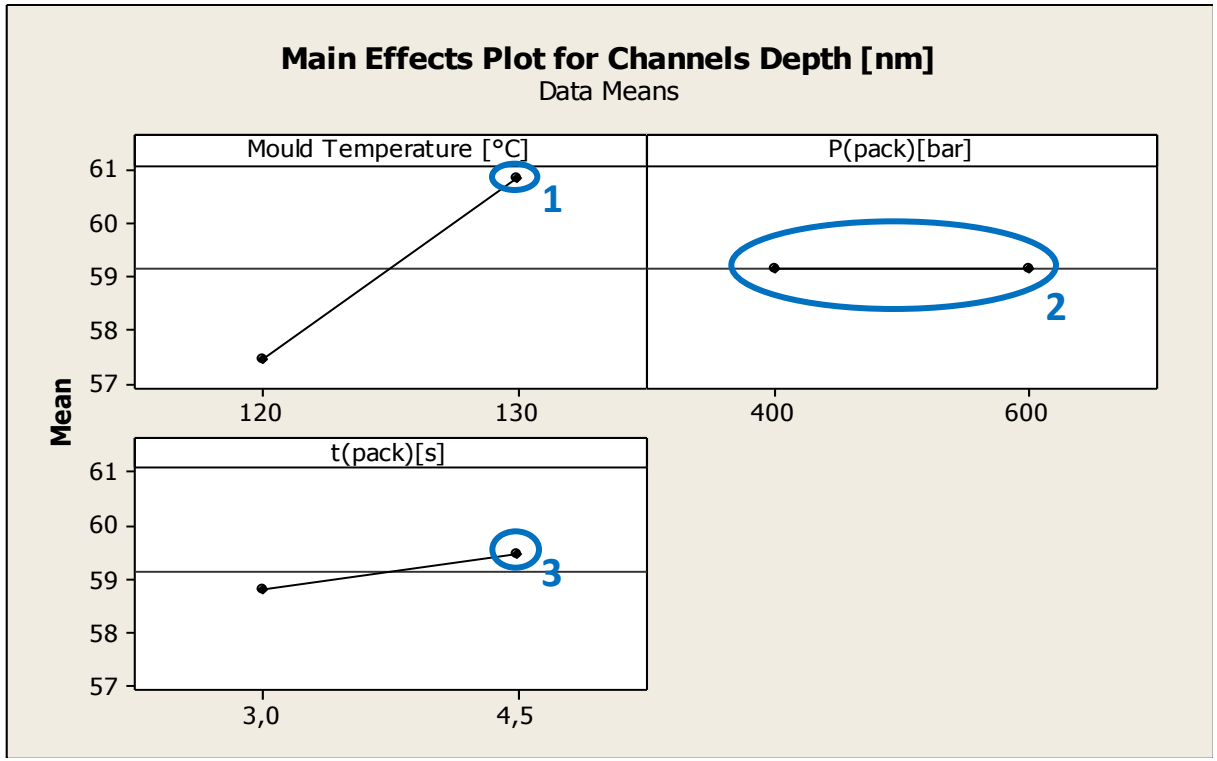
# Process optimization

## -Main effect plots-

1- Increasing mould temperature increases replication fidelity

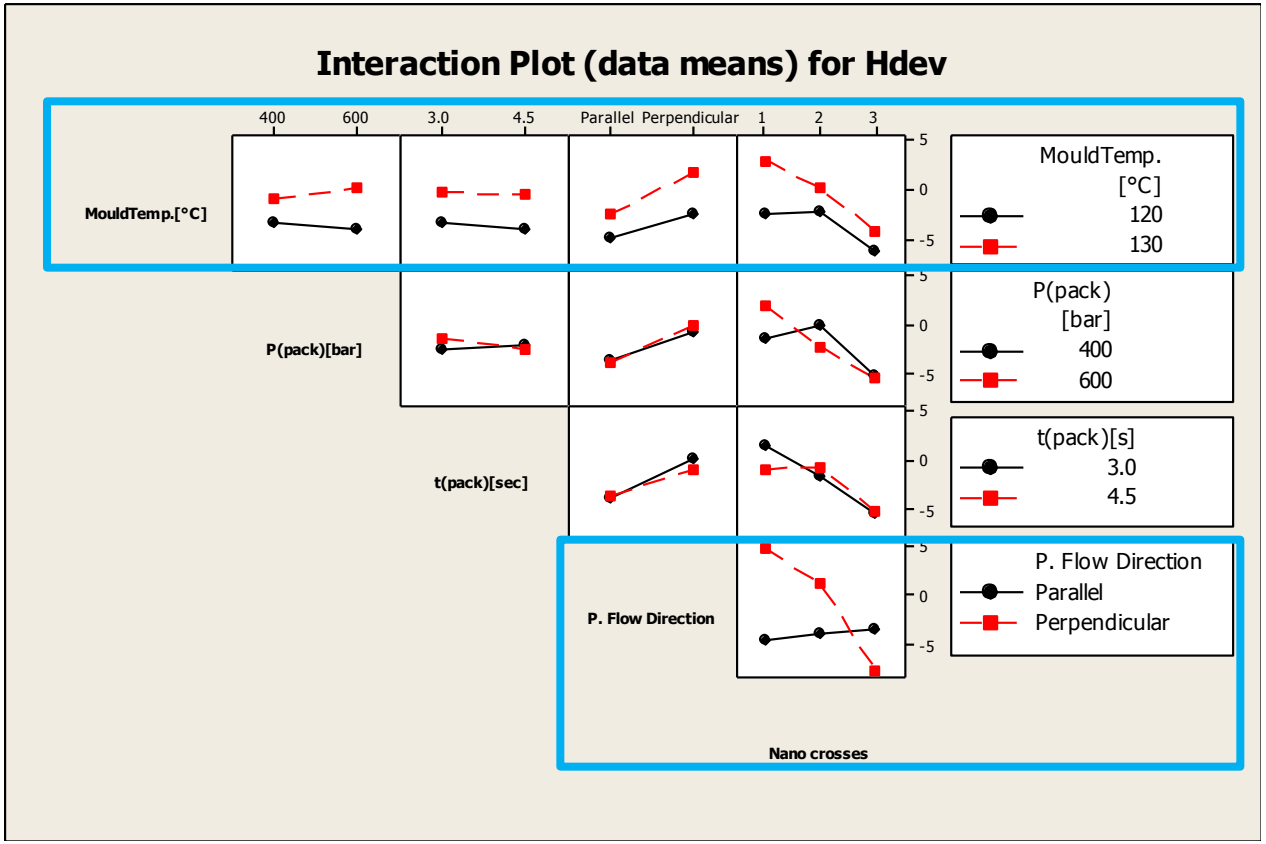
2- No effects within process window

3- Little effects on replication fidelity is given with longer packing time



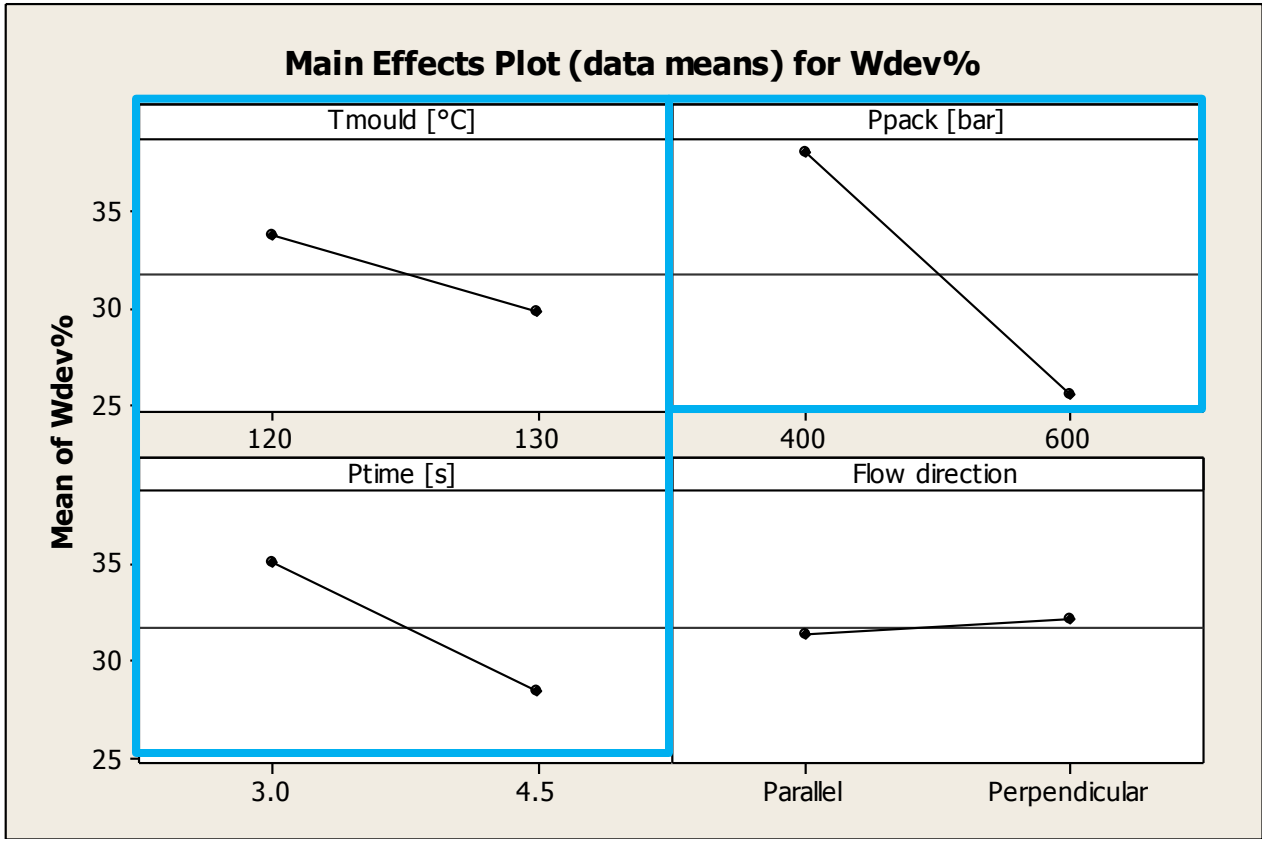
# Results and Discussion

$$Hdev = \left( \left| Polymer\ cross\ height \right| - \left| Nickel\ cross\ height \right| \right)$$



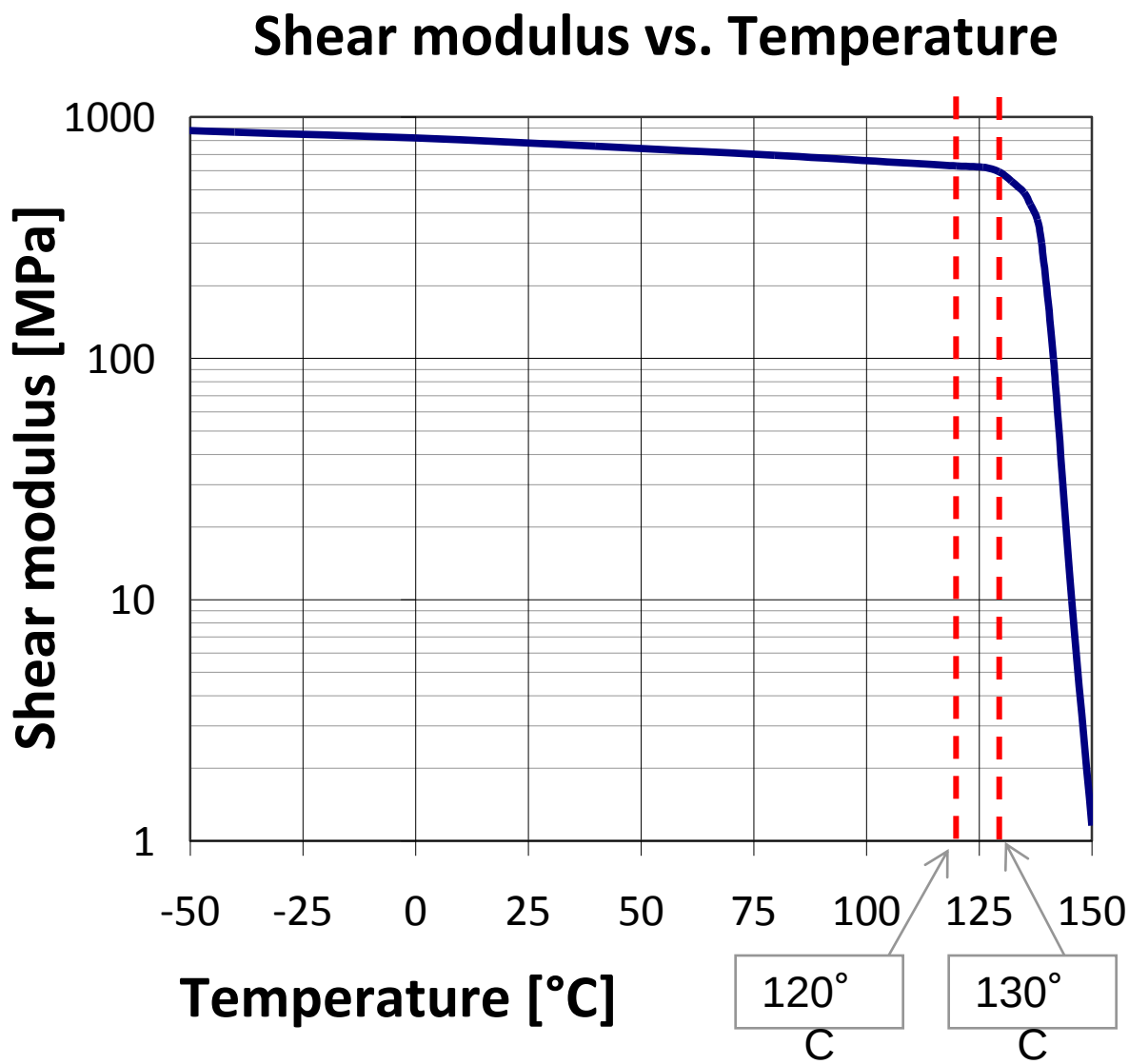
# Results and Discussion

$$Wdev\% = \left( \frac{(Polymer\ channel\ width - Nom.\ channel\ width)}{Nom.\ channel\ width} \right) * 100$$



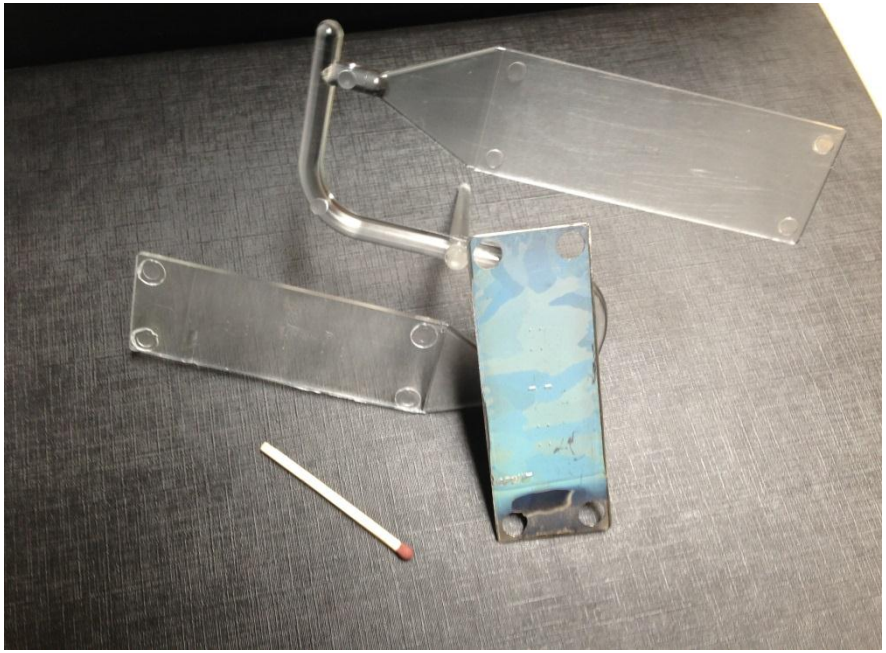
# Material characteristics

- Cyclic Olefin Copolymer
- COC TOPAS® 6013
- High heat deflection temperature
- Chemical resistance
- Bio/compatible
- Sterilization



# Experimental injection molding

## Patterning of periodic nano meter features



Process parameters

Mould temp. 130 ° C – Packing pressure 600 bar- Packing time 4,5 s

Melt temp. at 250° C and injection speed at 150 mm/s constant during experiments

# Mould fabrication

## Semi-spheres pattern

Electropolishing :
10 minutes; 85°C; 15 Volts;
$H_3PO_4$(80%)+$H_2SO_4$(20%)

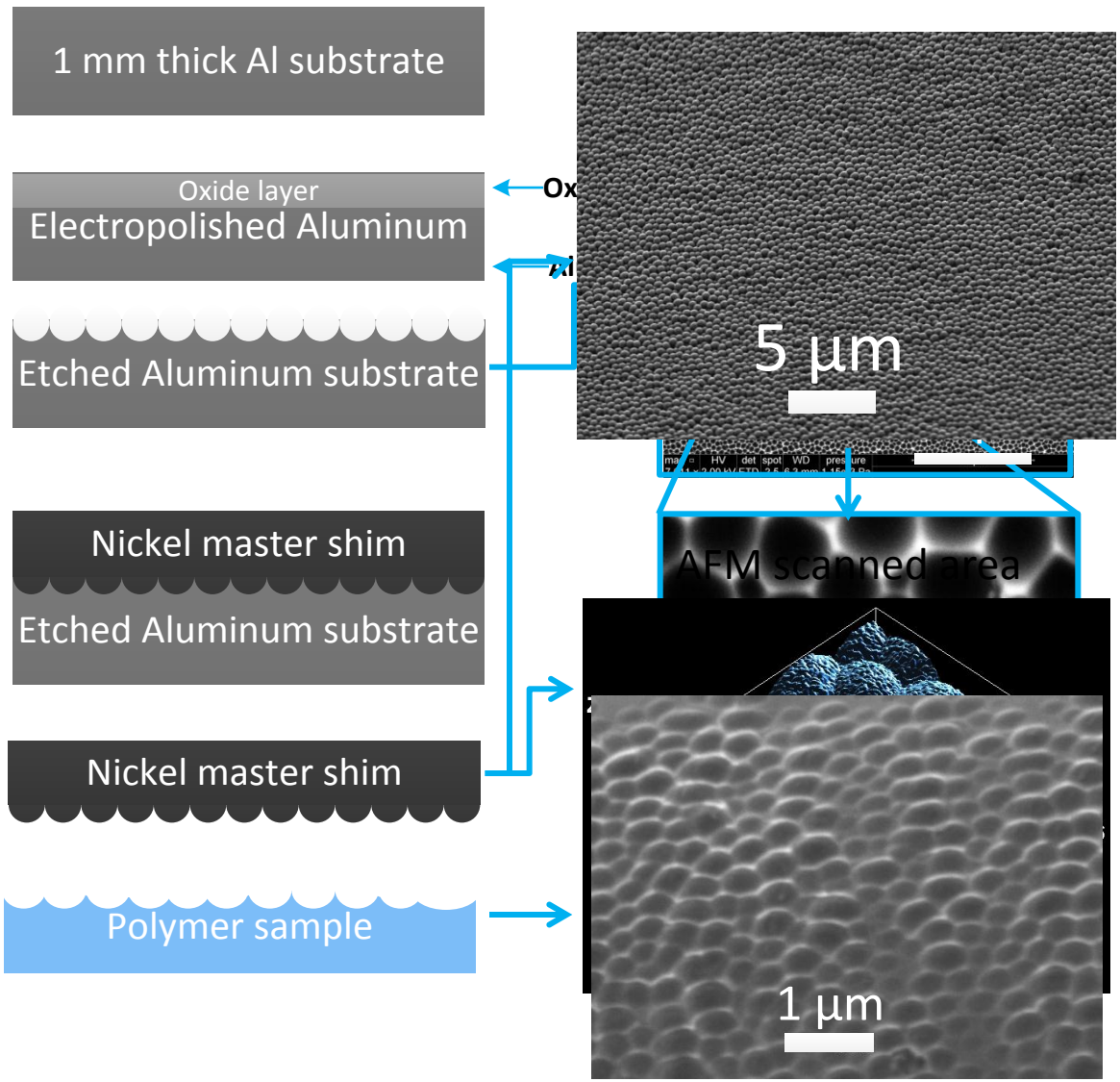
Anodization: 24 hours; 0°C;
195 Volts; $H_3PO_4$

Etching:24 hours; 85°C;
H3PO4 (35ml/liter)+$CrO_2$ (20g/liter)

Nickel vapor deposition and
Nickel electroplating

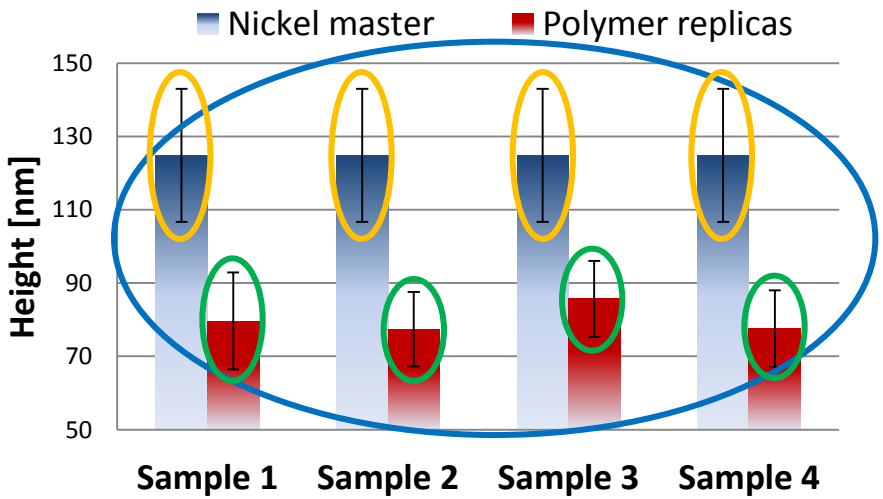
Selective etching
(Al dissolution)

Patterned injection moulded
polymer part



# Data analysis – Height

Height of semi-spheres



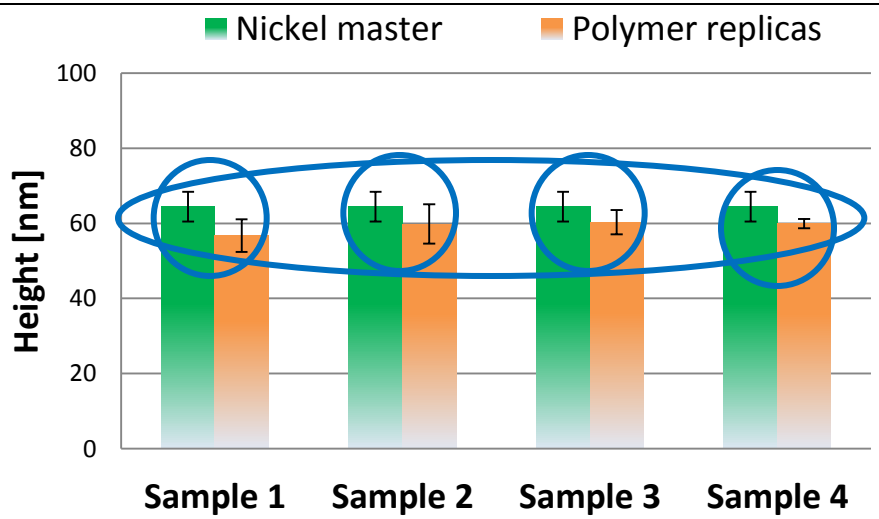

Error bars = average std. dev. of 10 meas.

1 - Full replication not achieved (≈65%)

2 - Std. dev. → polymer < nickel

→ contiguous features = polymer flow does not reaches the stamper base

Height of deter. structures




Error bars = average std. dev. of 10 meas.

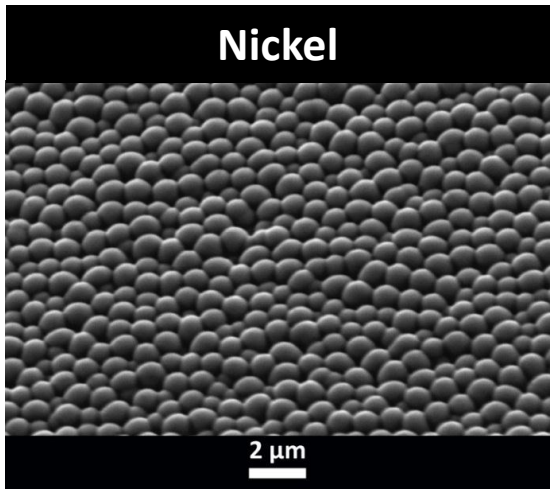
1 - High replication fidelity (≈95%)

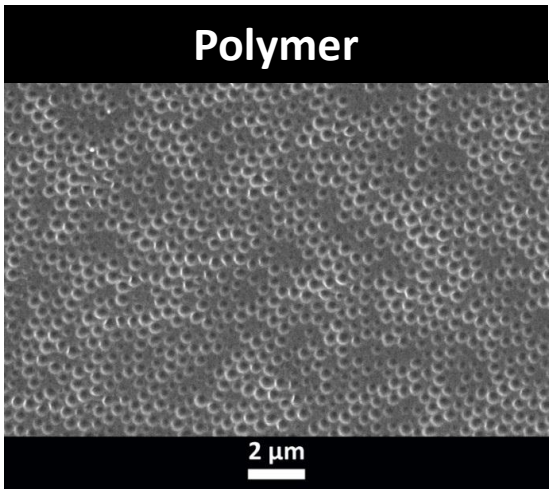
2 - Std. dev. → polymer ≈ nickel

→ no contiguous features = polymer flow reaches the stamper base

# SEM analysis – Surface sub-µm features

SEM qualitatively control

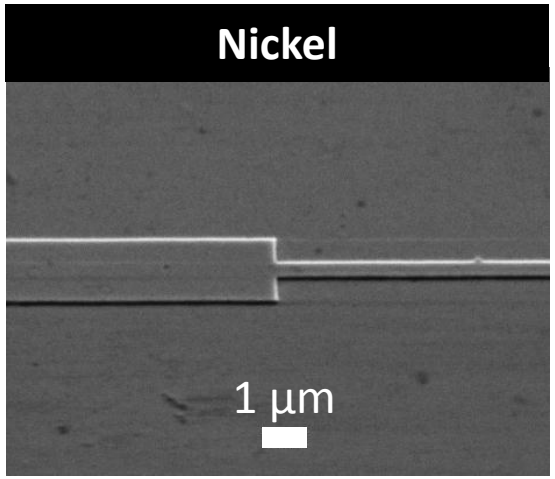


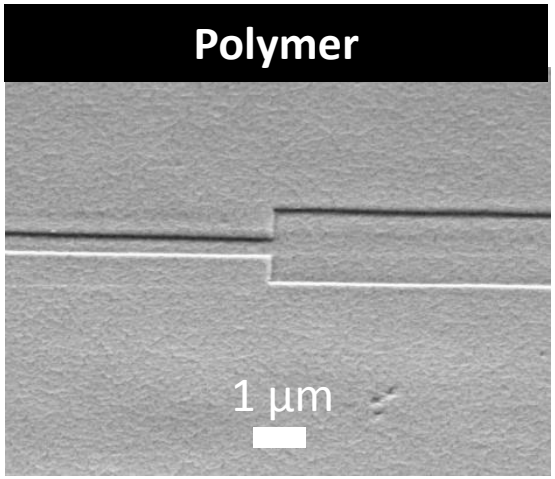




1- Full replication not achieved

2- Standard deviation calculated on polymer samples smaller than corresponding nickel geometries

SEM qualitatively control



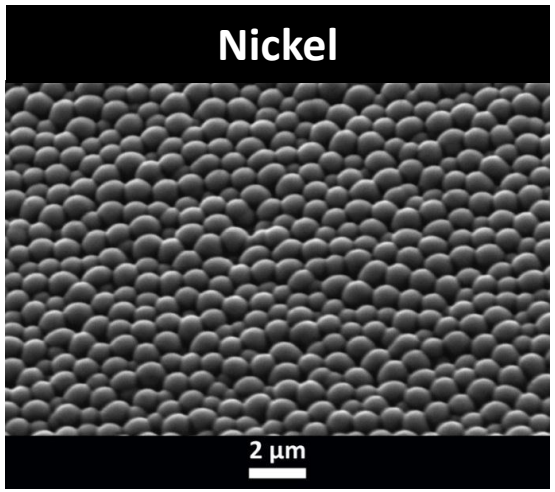



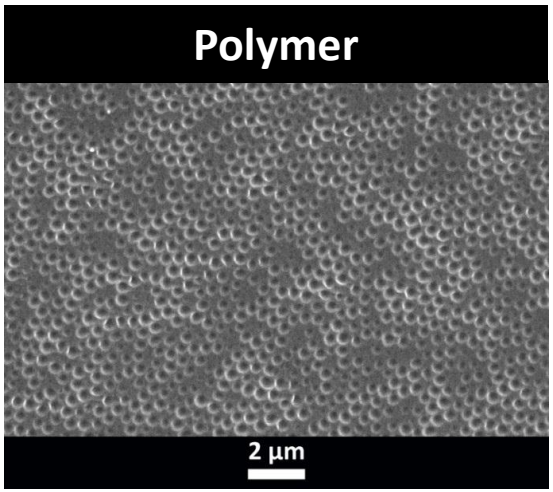

1- High replication fidelity

2- Standard deviation in the same order of magnitude → no contiguous features = polymer flow reaches the stamper base

# Surface sub-µm features width

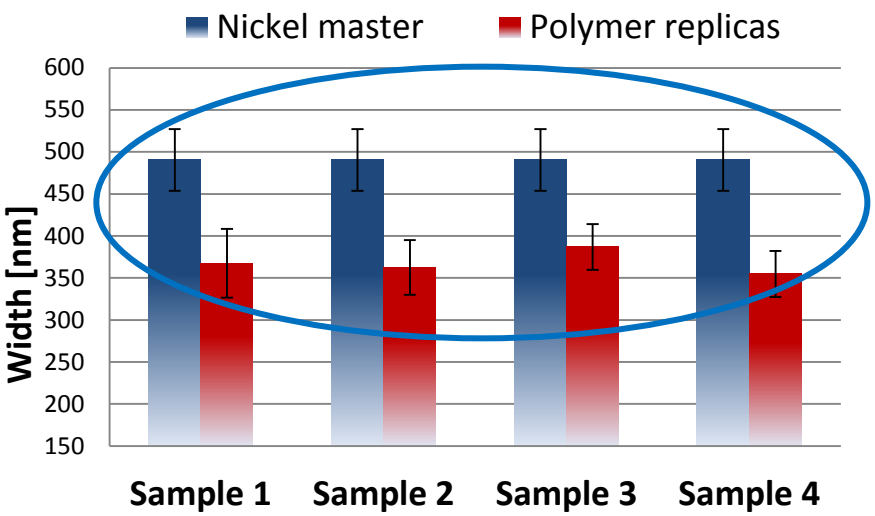
SEM qualitatively control



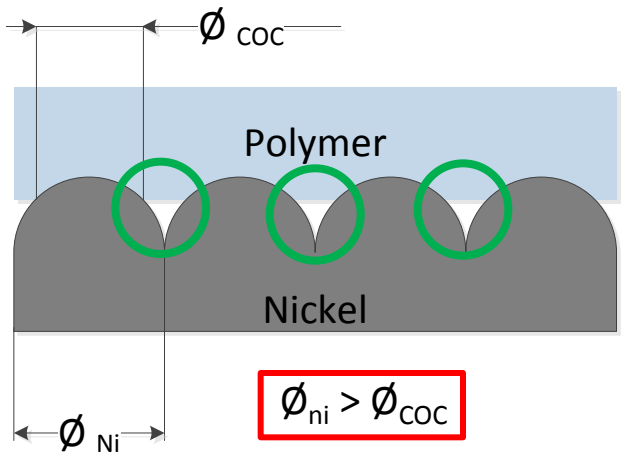




Full replication not achieved

Width of semi-spheres



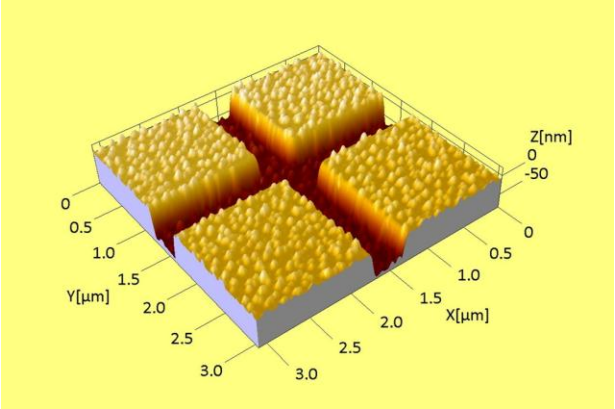

Error bars = average std. dev. of 10 meas.



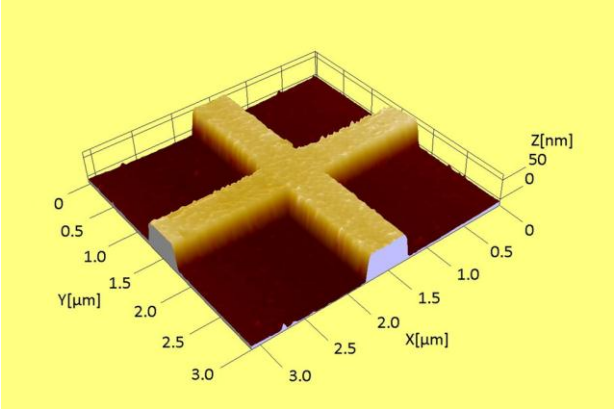
# Tolerance Verification at nm-scale

## Product compliance with specification

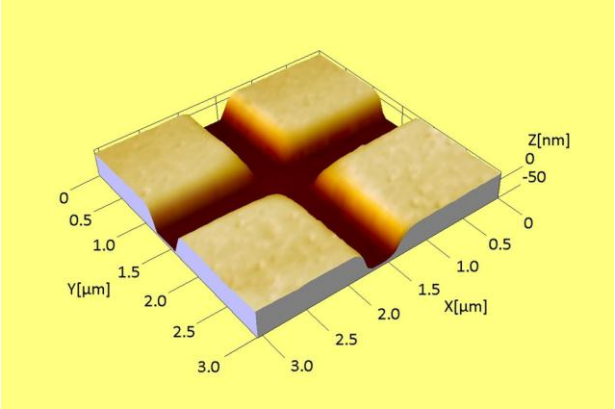
Quality control ↔ Process chain capability (Uncertainty) ↔ Tolerance limits




Etched silicon master geometries



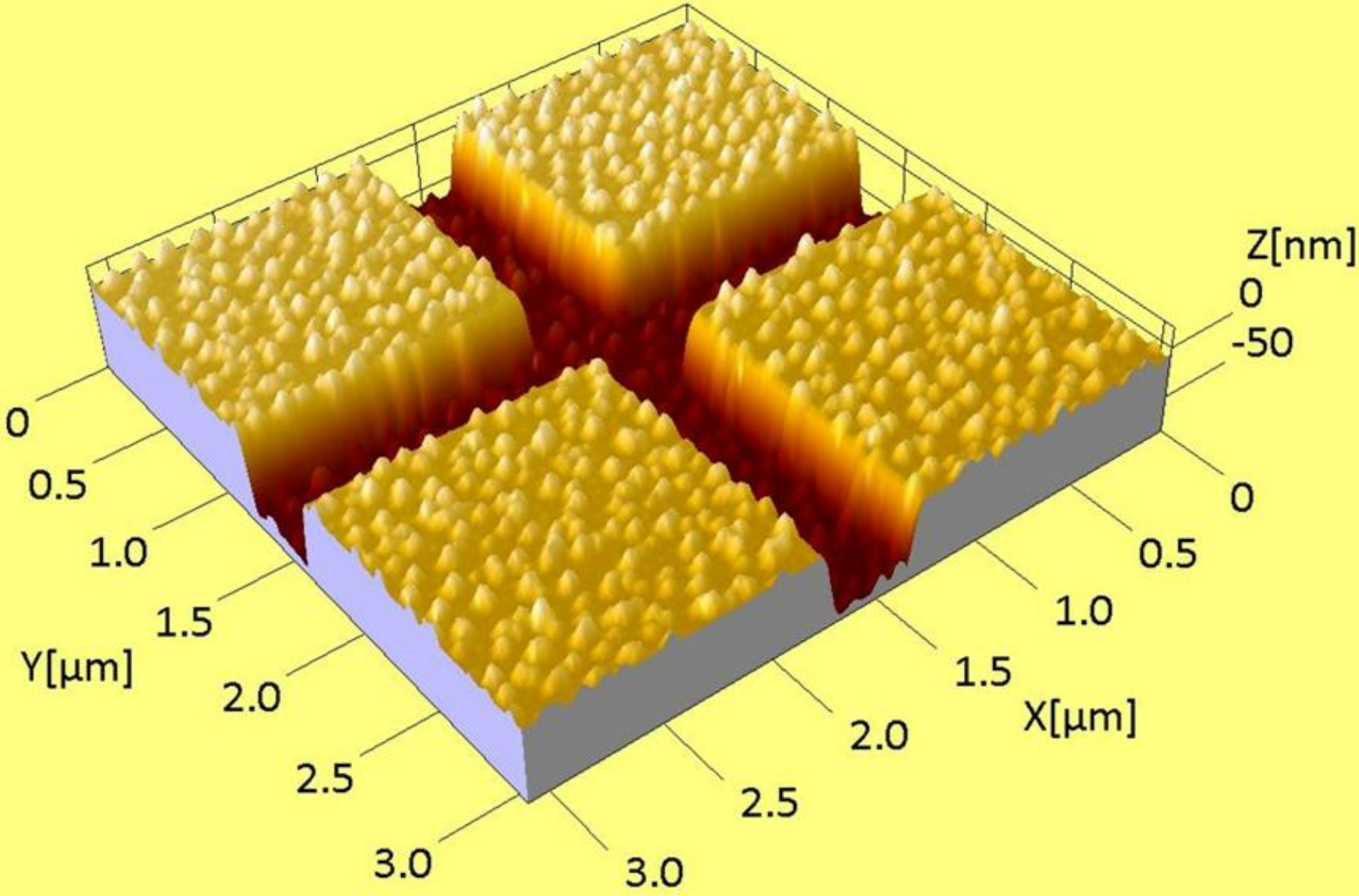

Nickel electroplated geometries



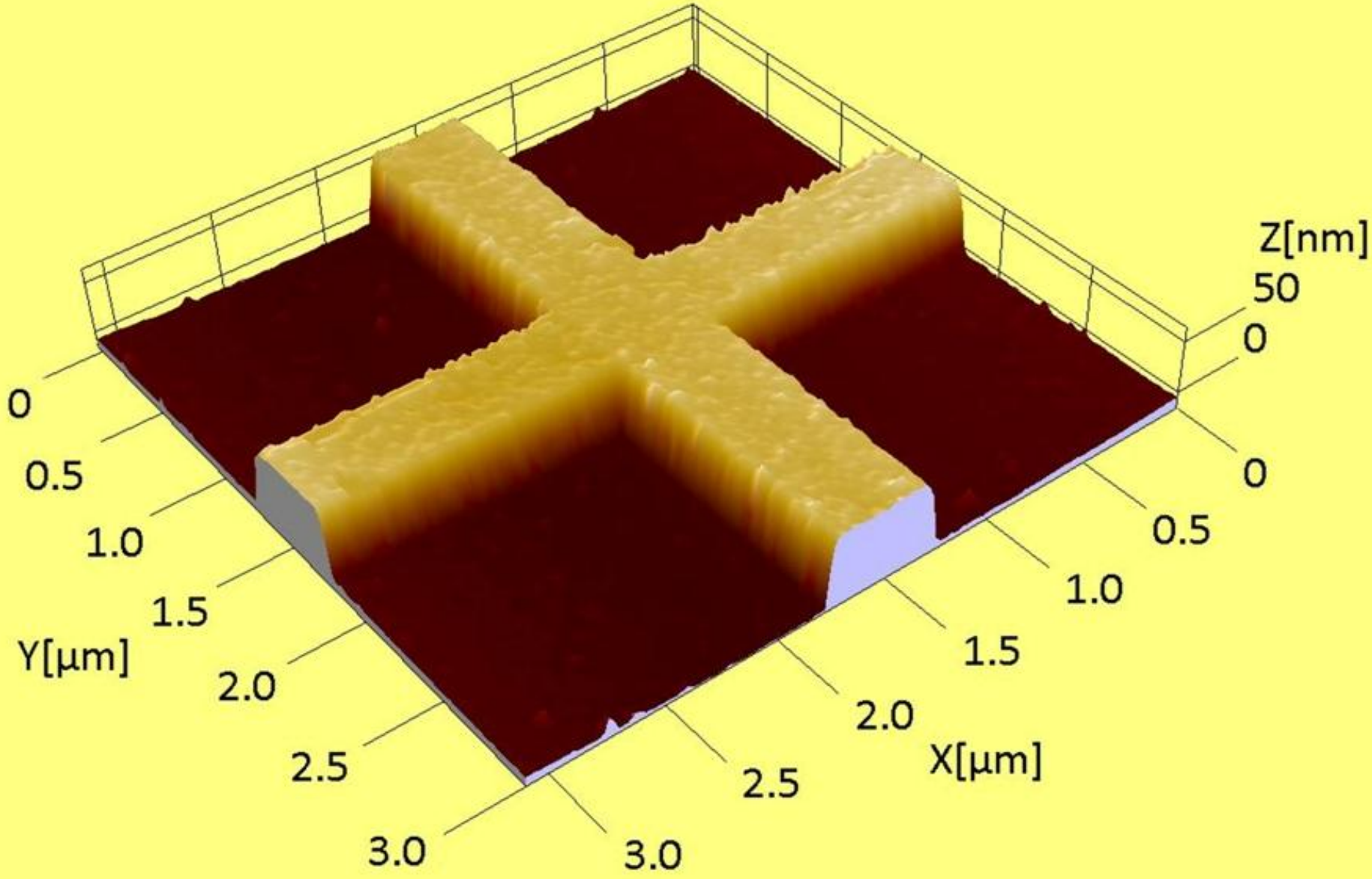

Injection moulding replica

AFM measurements (magnification: X 1x, Y 1x, Z 5x)

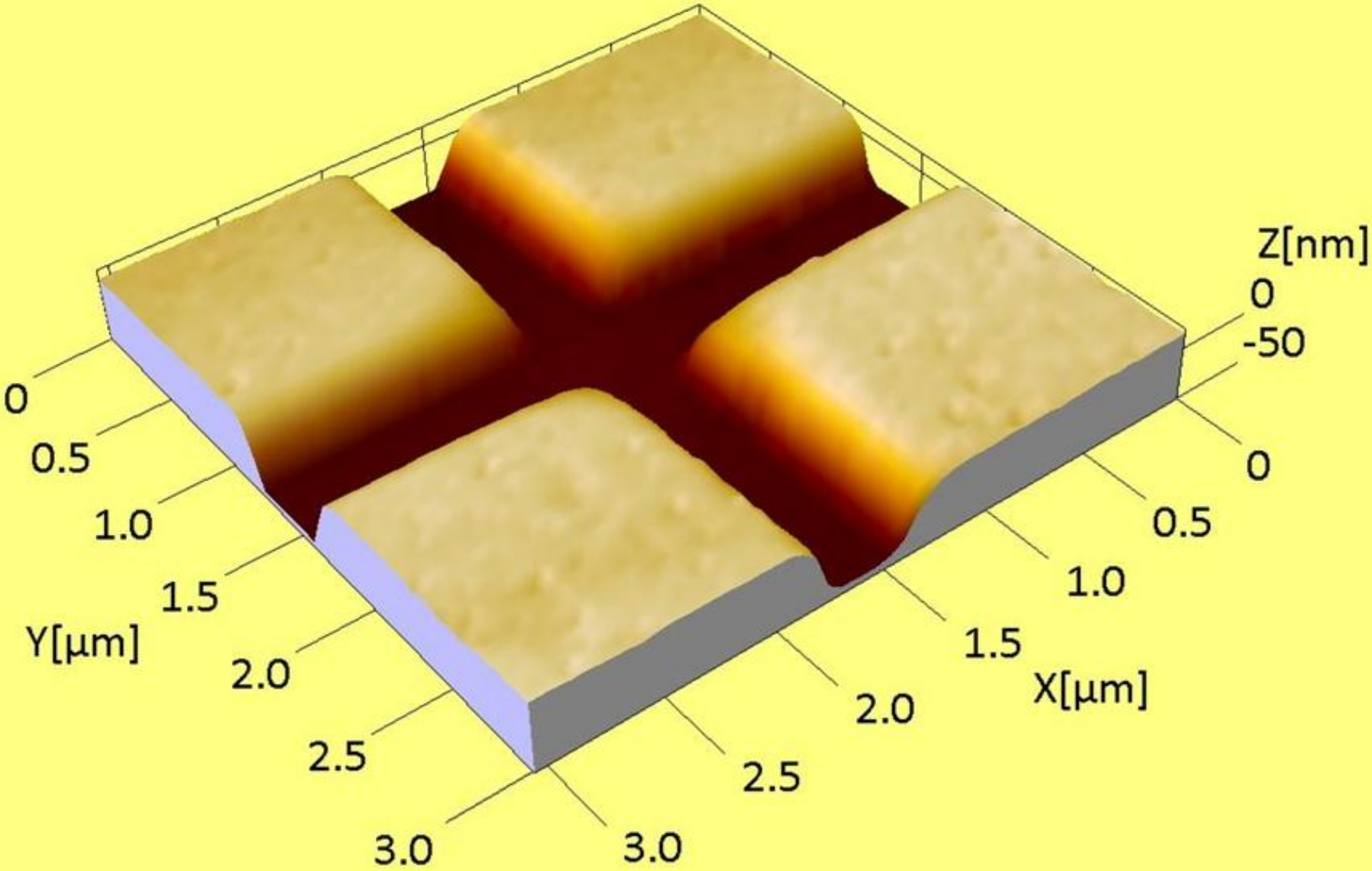
## E-beam lithography of silicon



**Nickel electroplating**



**Micro injection moulding (COC Topas® 6015)**



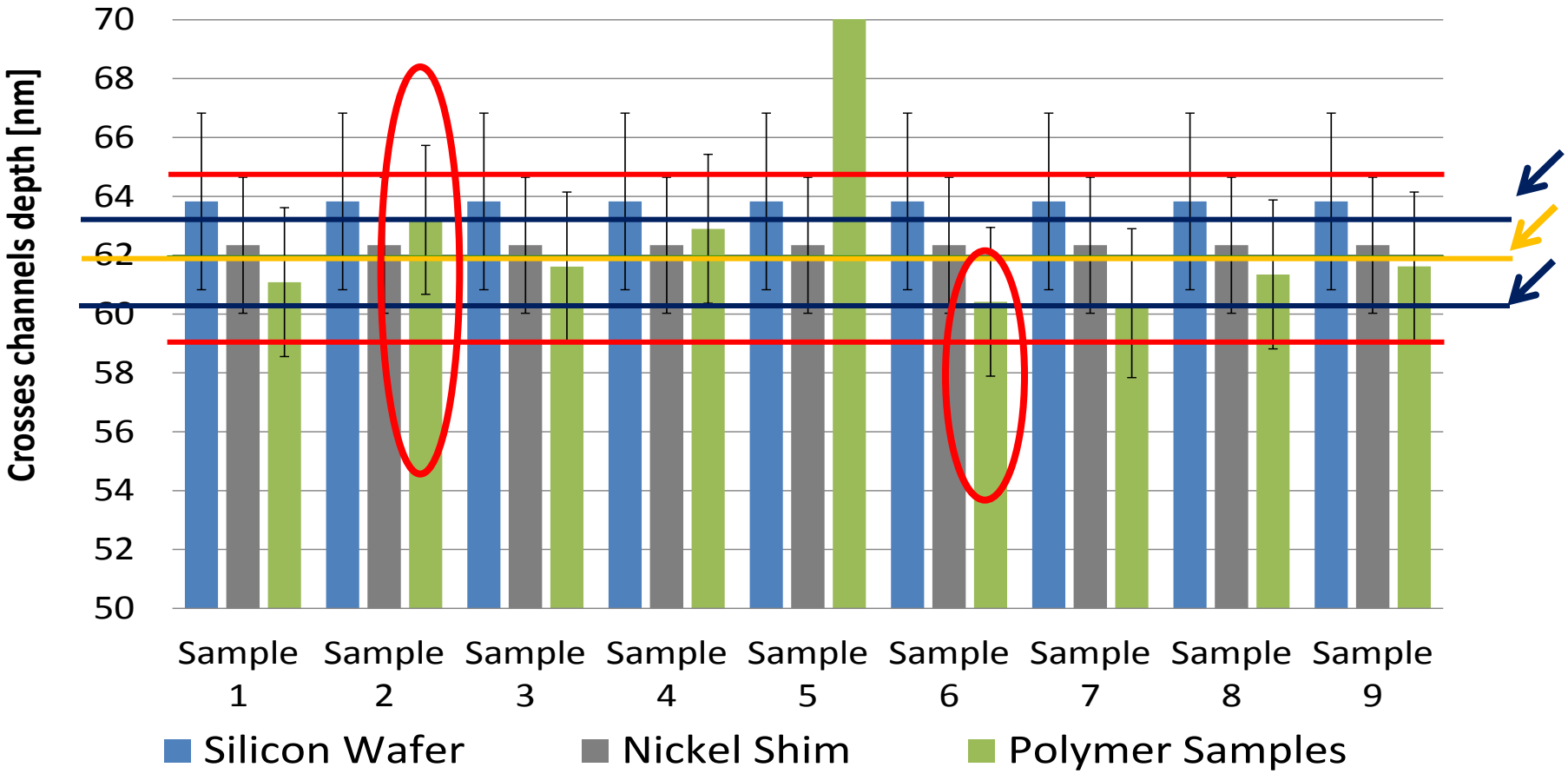
# Uncertainty assessment

Uncertainties of the Z-measurements, based on ***ISO 5436*** profile analysis, were calculated following the ***GUM***

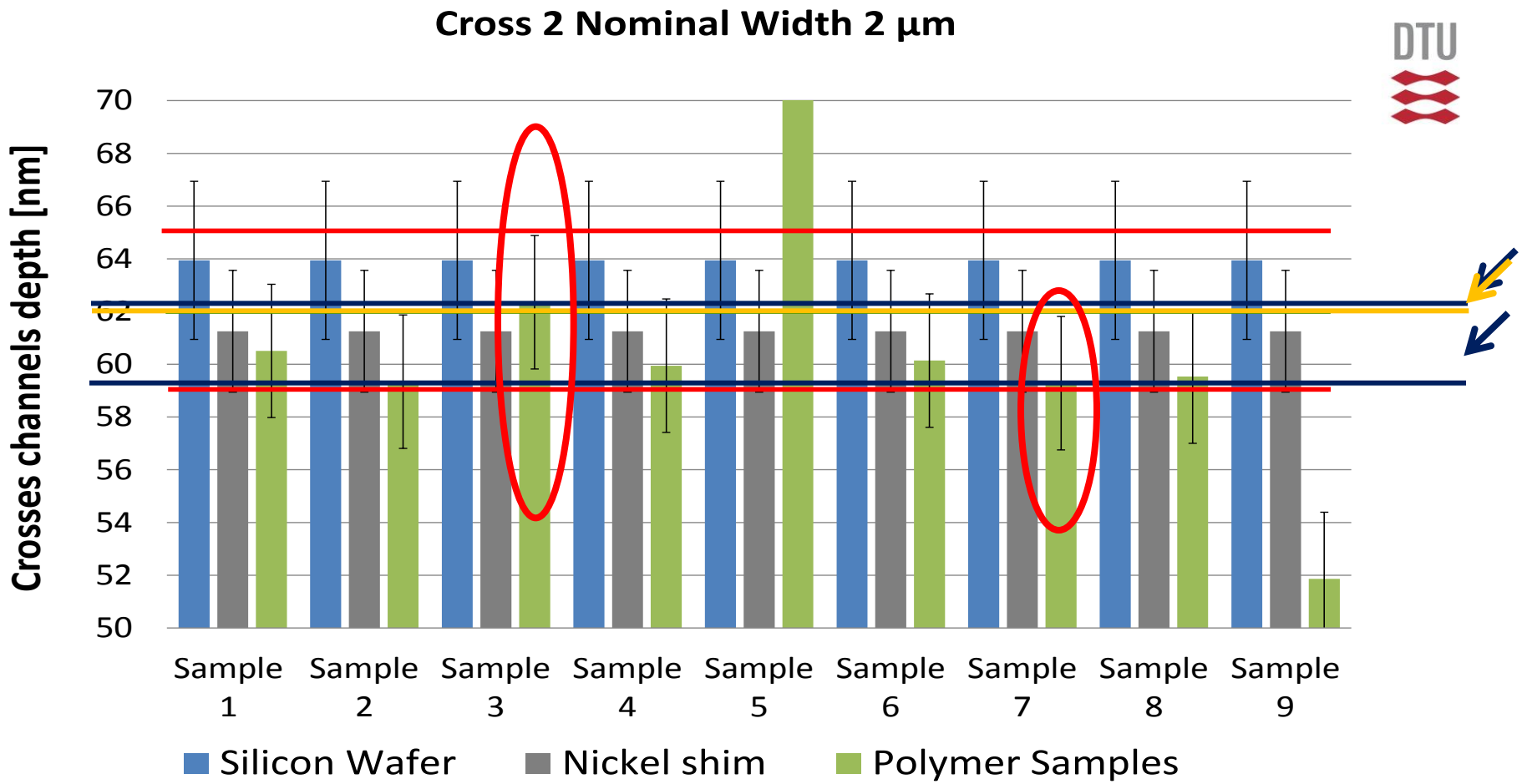
$$U_i = k \cdot \sqrt{u_{res,z}^2 + u_{res,c}^2 + u_{cal,z}^2 + u_{rep,z}^2 + u_{noise}^2}$$

| ***$u_j$ contributor*** | ***Silicon Wafer*** | ***Nickel Shim*** | ***Polymer Samples*** |
|---|---|---|---|
| $u_{res,z}$ (vertical resolution) | 0,05 | 0,05 | 0,05 |
| $u_{c,z}$ (calibration factor) | 0,58 | 0,59 | 0,60 |
| $u_{cal,z}$ (calibration artefact) | 0,75 | 0,75 | 0,75 |
| $u_{AFM,z}$ (instr. repeatability) | 0,43 | 0,42 | 0.36 |
| $u_{features,z}$ (feat. repeatability) | 0,97 | 0,50 | 0,35 |
| $u_{sample,z}$ (samp. repeatability) | 1,08 | 0,33 | 0,69 |
| $u_{noise}$ (background noise) | 0,43 | 0,43 | 0,43 |
| *U* (exp. comb. uncertainty) | 3,0 | 2,3 | 2,5 |

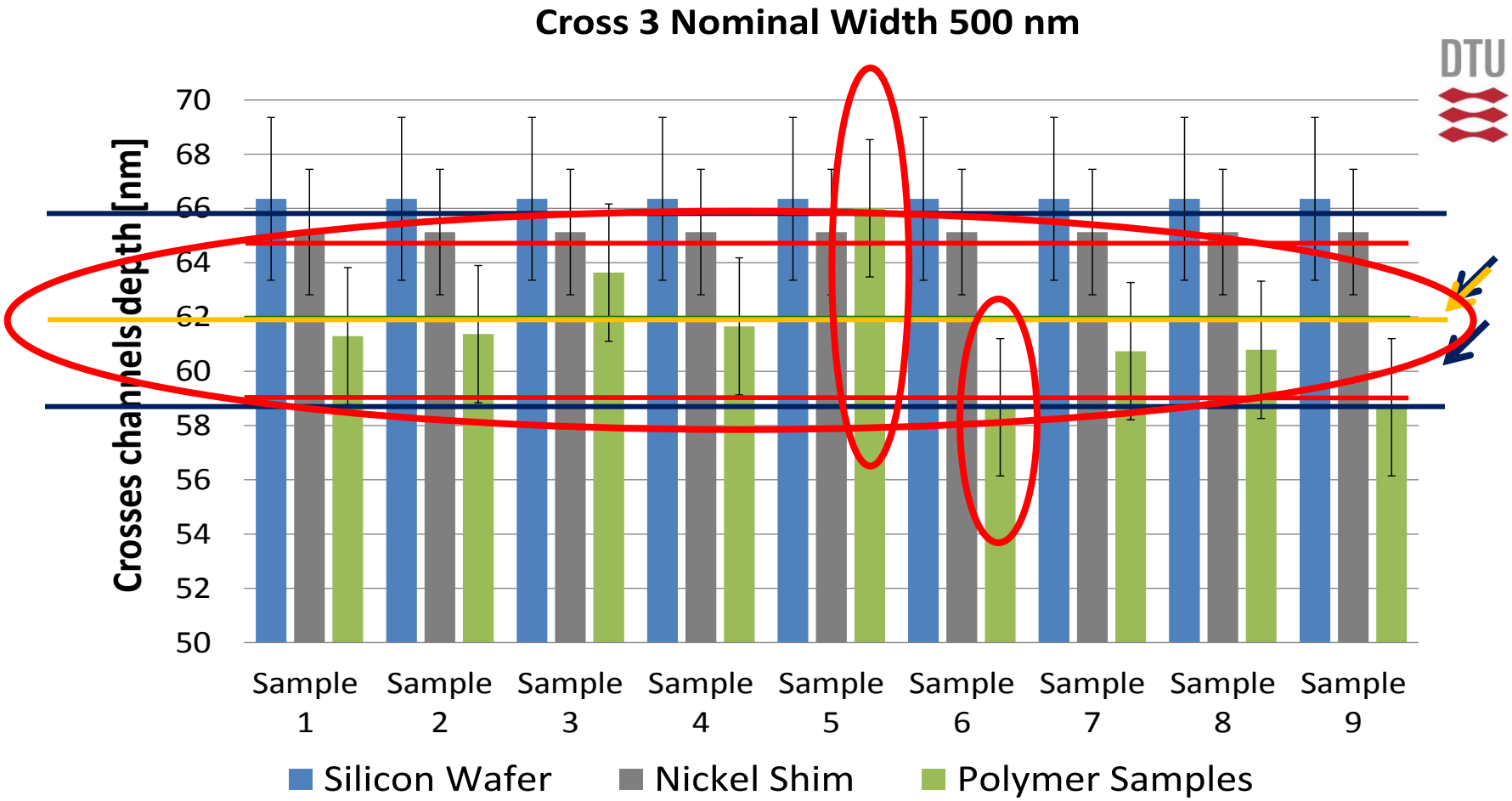
# Cross 1 Nominal Width 10 µm




- Largest depth deviation among the different polymer sample is equal to 3nm
- Average polymer channel depth replication fidelity equal to 98%




- Largest depth deviation among the different polymer sample is equal to 3nm
- Average polymer channel depth replication fidelity equal to 97%




- Feature variation exceeds the tolerance range
- Largest depth deviation among the different polymer sample is equal to 7nm
- Average polymer channel depth replication fidelity equal to 94%

# Conclusion

- Tooling ⇨ functional surface geometries in the nanometer range
- Moulding ⇨ deterministic and distributed nano features
- Quality control ⇨ calibrated AFM measurements
- Replication fidelity
    - Positive (master) and negative (polymer)
    - ⇨ height/depth
    - ⇨ width
- Tolerance verification at nm/scale

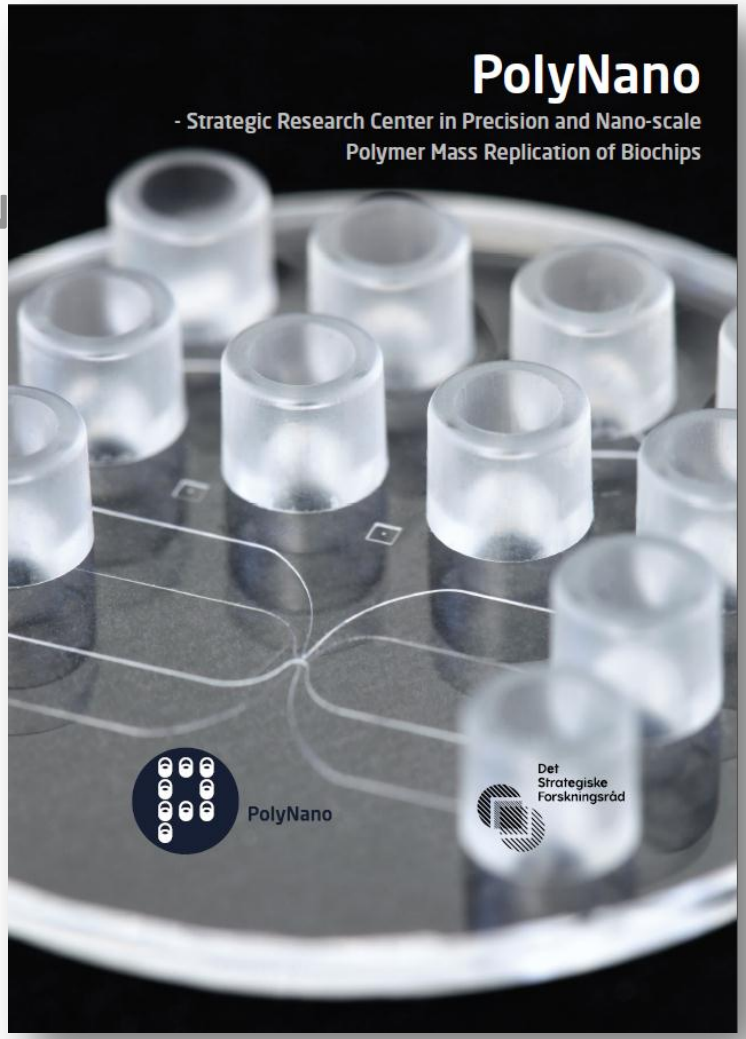
# Outlook

- Development of polymer nano manufacturing standard verification
  - Method ⇨ high precision, fast, reliable, traceable
- Tolerance dimensional range = ≤10-20 nm
- Measurements uncertainty range 1-5 nm needed
- Micro to nano scale structure integration
- In-line capability not yet available

# Acknowledgements

## PolyNano

- Strategic Research Center in Precision and N
  Replication of Biochips (2011-2016)
- Danish Council for Strategic Research
- http://www.polynano.org/



# *Thank you for your kind attention*

## Surface replication of sub-µm structures
## Analysis, optimization and quality assurance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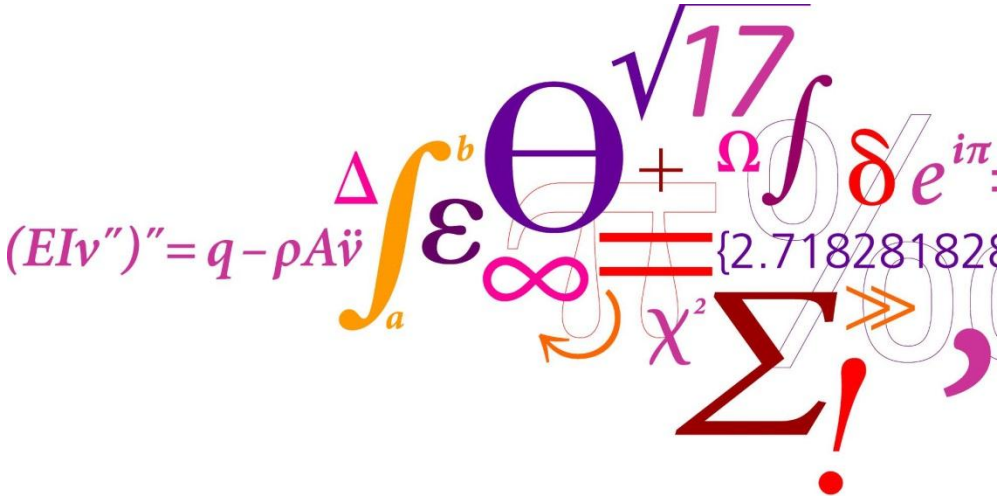

# Process control monitor