

Bulk Material Micro-structuring and Surface Modifications

Micro-structuring

mechanical

electrical (EDM)

chemical

irradiation

electrochemical

Surface modification

electrodeposition

PVD/CVD

polymer coatings

chemical

SAMs

Materials

metals

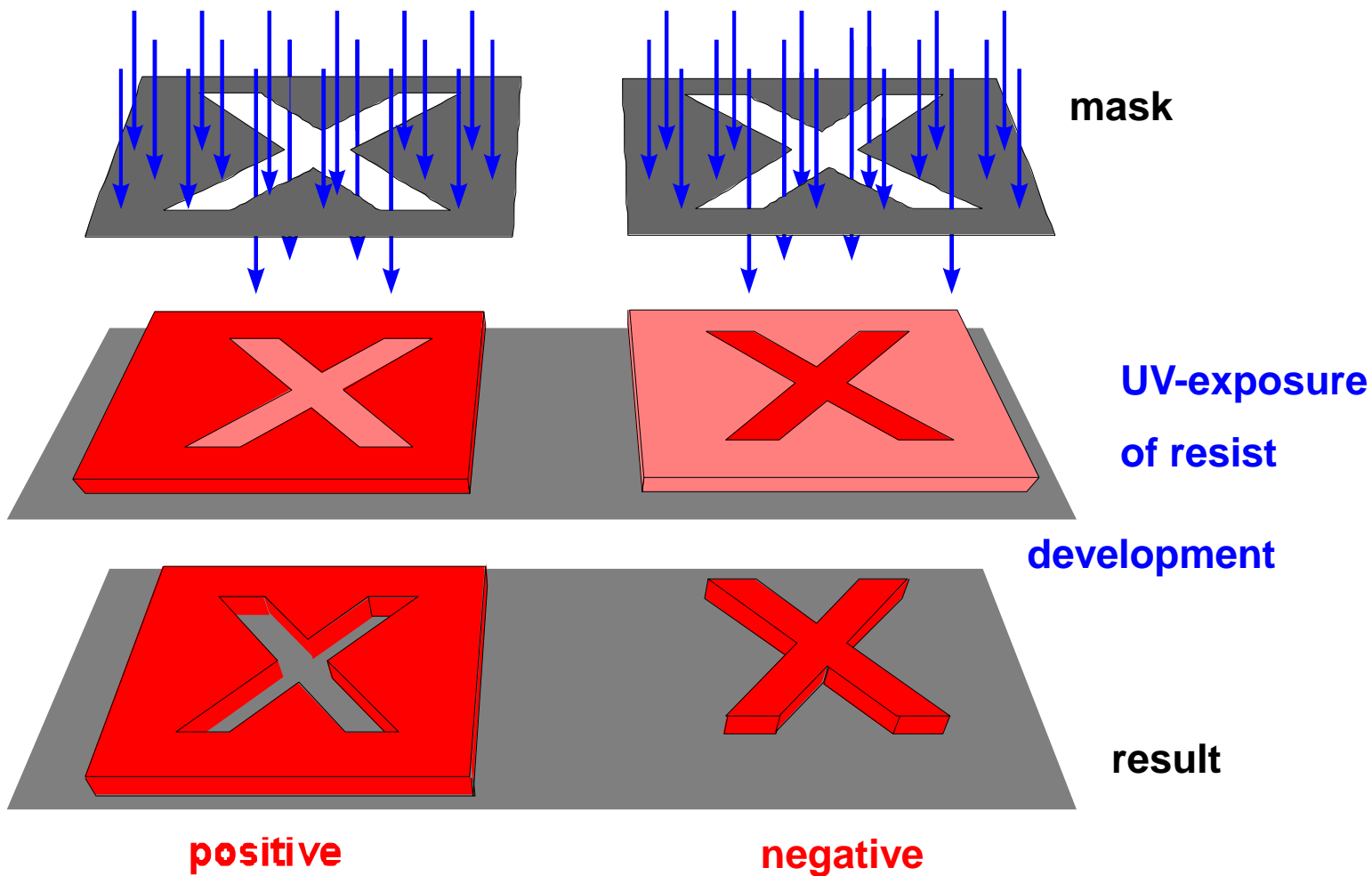
ceramics

glass

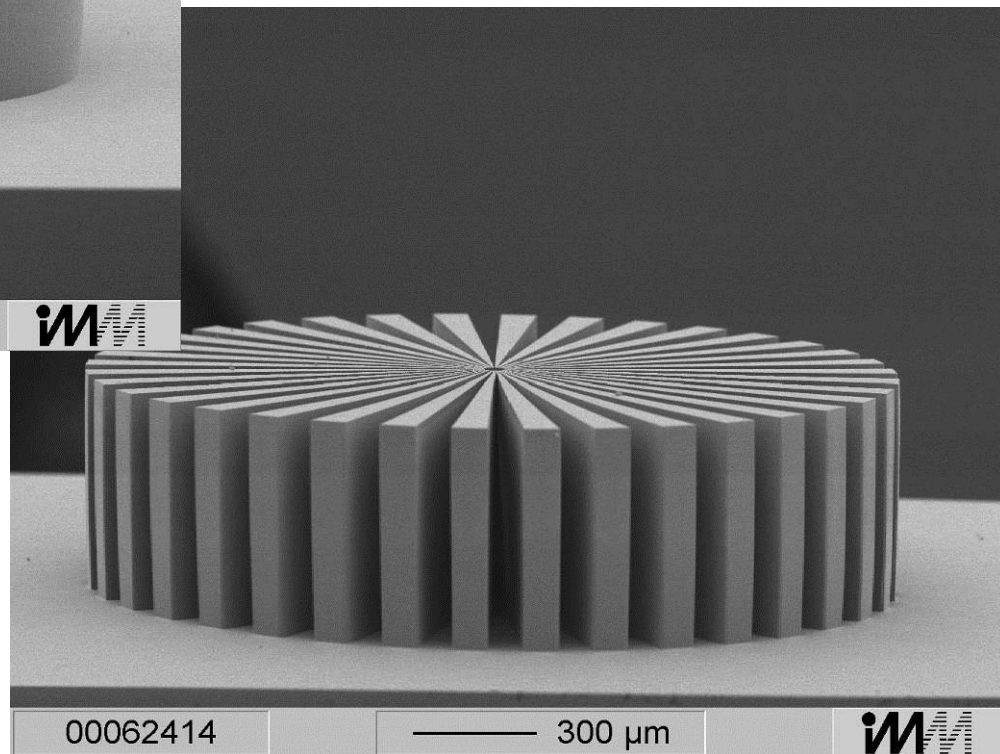
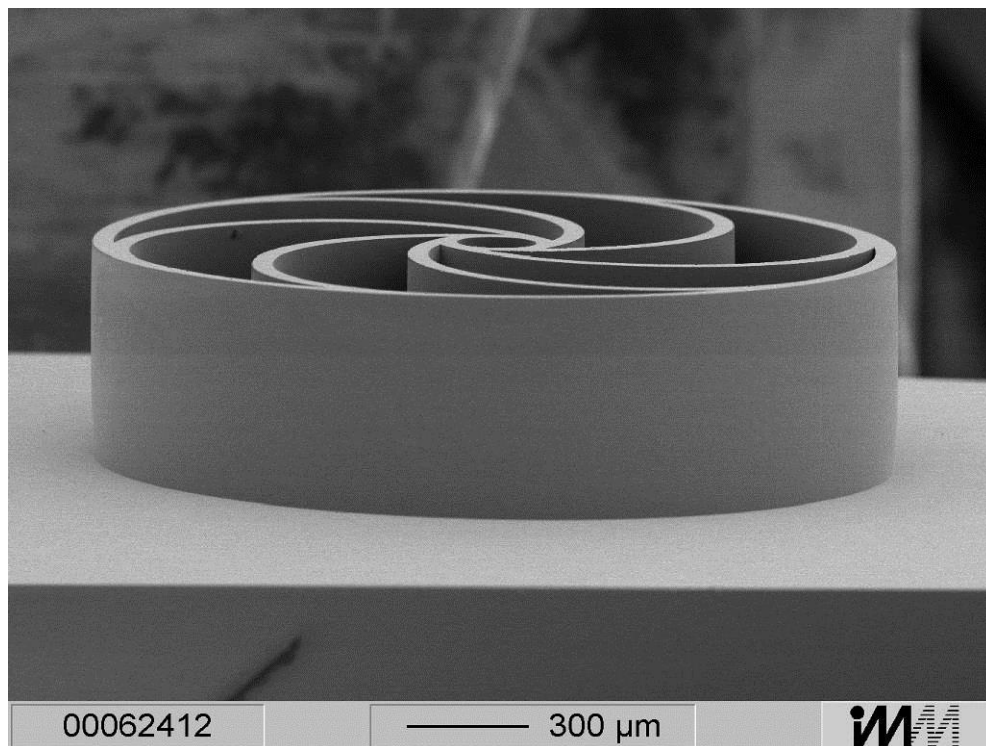
semiconductors

polymers

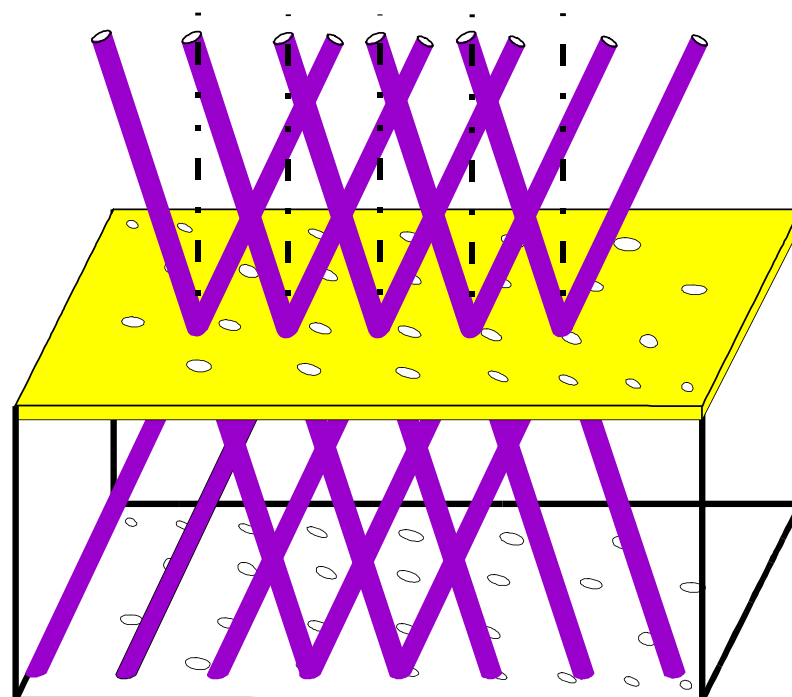
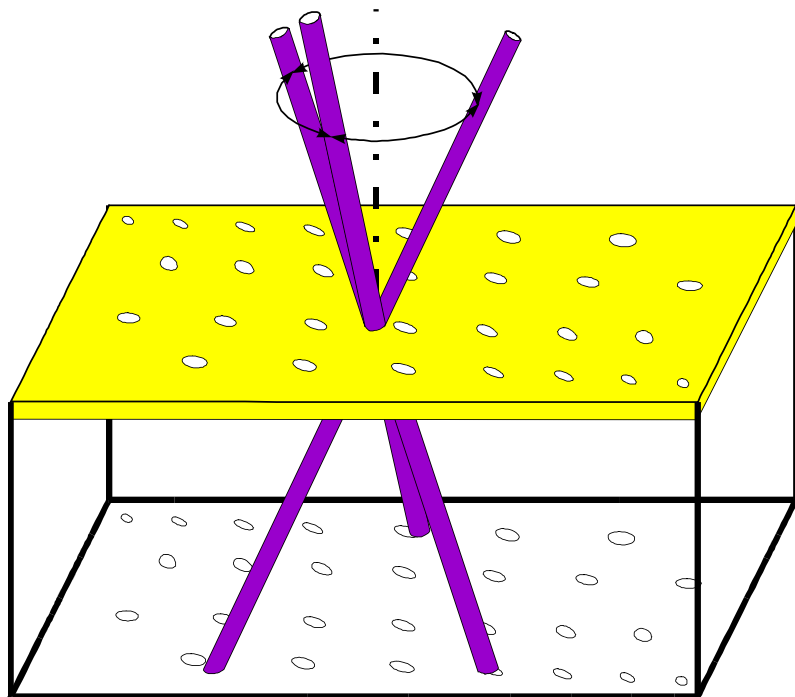
Principle of Optical Lithography



SU-8 Microstructures to be Filled by Electroforming



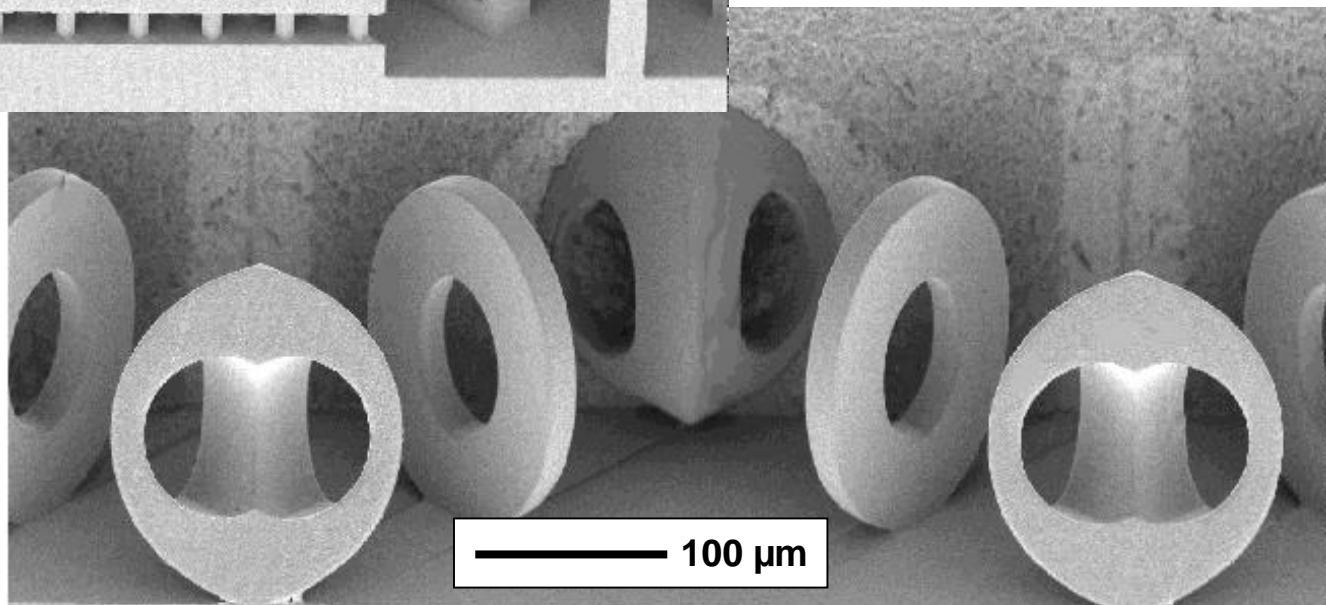
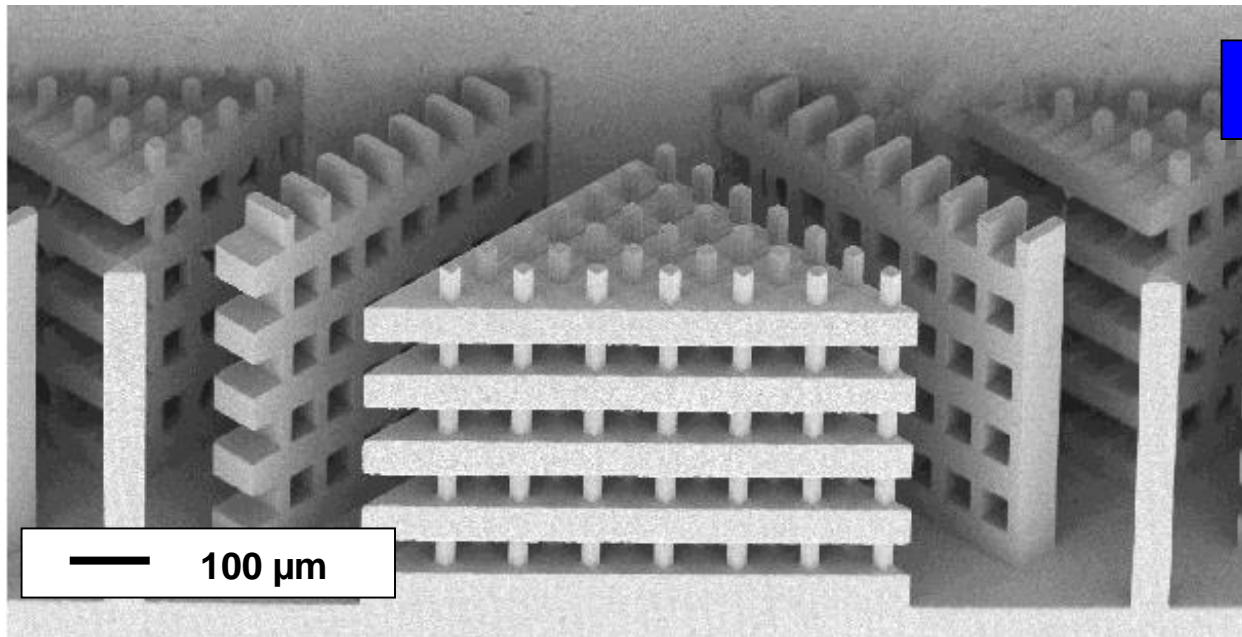
Fabrication of Photonic Band Gap Structures by X-ray Lithography



**Generation of a fcc type lattice structure
by triple inclined irradiation**

3-D Microstructures in PMMA

Made by LiGA - technique

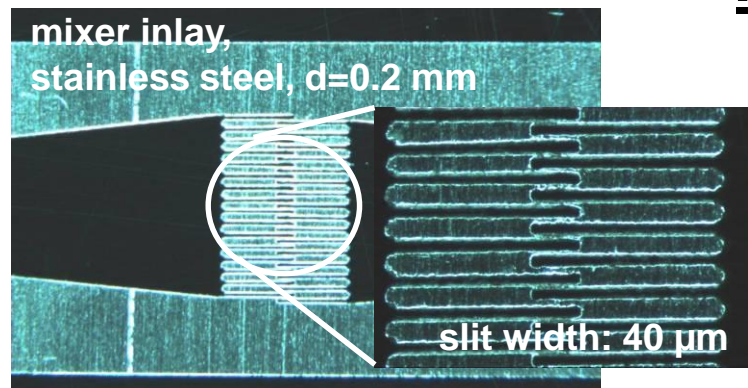


Nd:YAG Laser Micromachining

Applications:

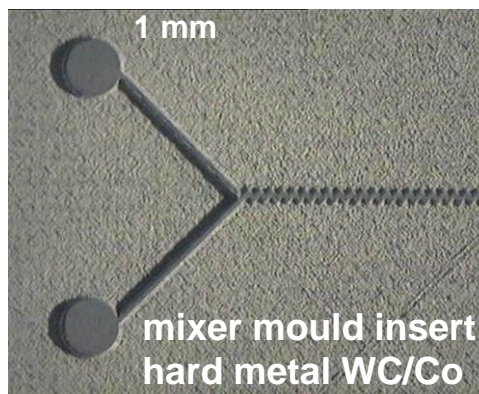
Cutting & Drilling:

- sublimation cutting, small kerf width
- material: metals, ceramics silicon, polymers (filled)
- small hole diameters (>10 μm , aspect ratio <100)



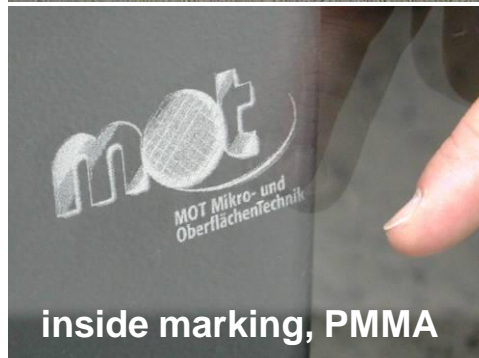
Workstations:

Laser: Nd:YAG (doubled)
Wavelength: 1064 (532) nm
Pulse length: 100 ns
Pulse energy: 10 (1) mJ, max.
XYZ-table, 10 μm accuracy
Scanner, feed rate up to m/s



Laser Milling:

- rapid tooling/prototyping
- material: metals, ceramics silicon, polymers (filled)
- high resolution (down to 30 μm structure size)



Laser Engraving:

- fast marking
- polymer inside marking
- high processing speed

Eximer Laser Micromachining



EXITEC-Workstation:

Laser: Excimer LPX110i

Wavelength: 193 nm

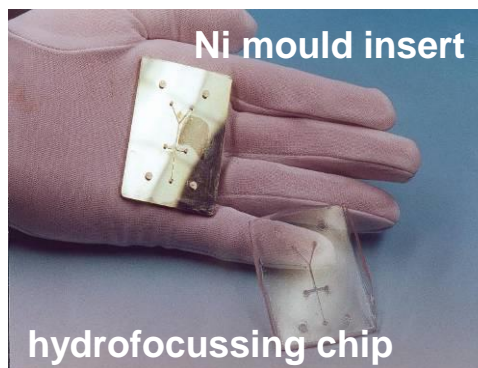
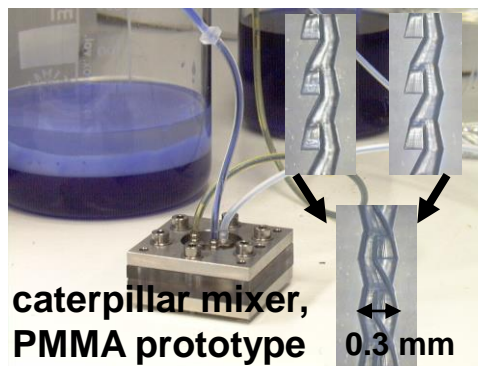
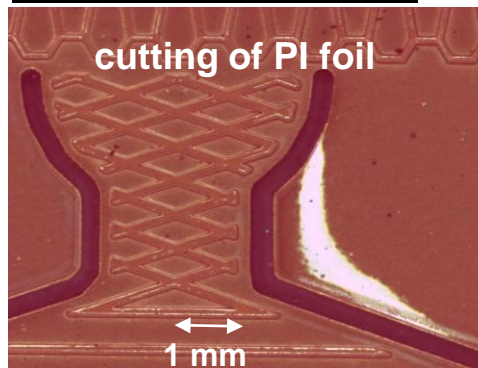
Pulse length: 17 ns

Pulse energy: 200 mJ (max.)

XYZ-table, 1 μm accuracy

Mask projection

Applications:



Polymer Machining:

- ablation (depth 5 μm to 1 mm, resolution $<5 \mu\text{m}$ $R_a > 0.2 \mu\text{m}$, quasi 3D)
- fine cutting
- drilling (various hole shapes)

Rapid Prototyping:

- microfluidic channels
- polymer bio chips
- microoptical structures

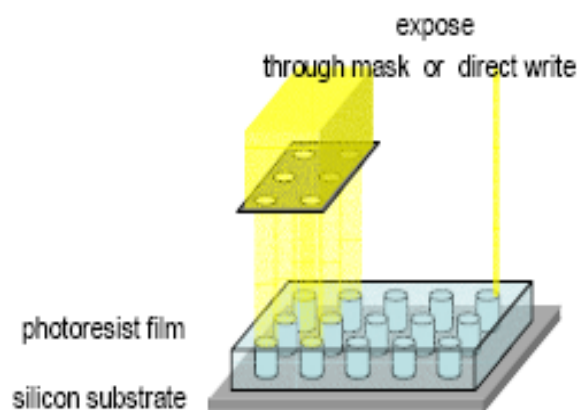
Laser LIGA:

- mould insert fabrication
- material: Ni, Cu
- large number production (hot embossing, injection moulding)

Direct Laser Writing: Introduction

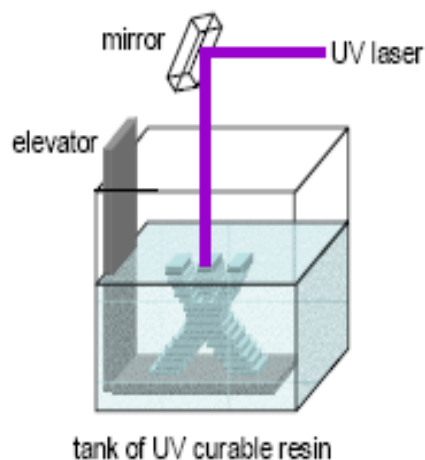
Conventional Lithography

- structures are 3-D representations of 2-D images
- spatial resolution of 20 nm – 1 μm depending on radiation source:



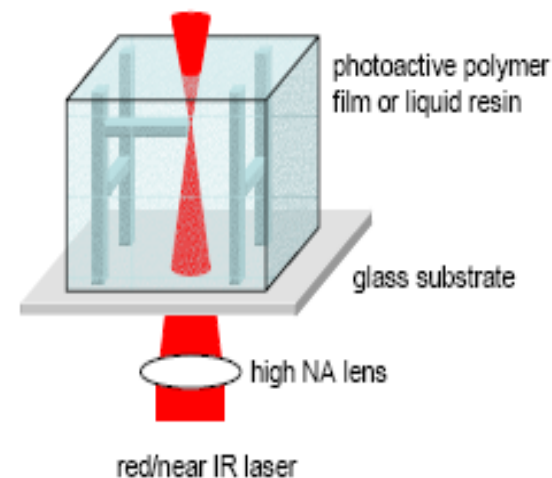
Micro Stereolithography

- one-photon process
- 3-D structures
- layer-by-layer additive process
- spatial resolution of $>10 \mu\text{m}$

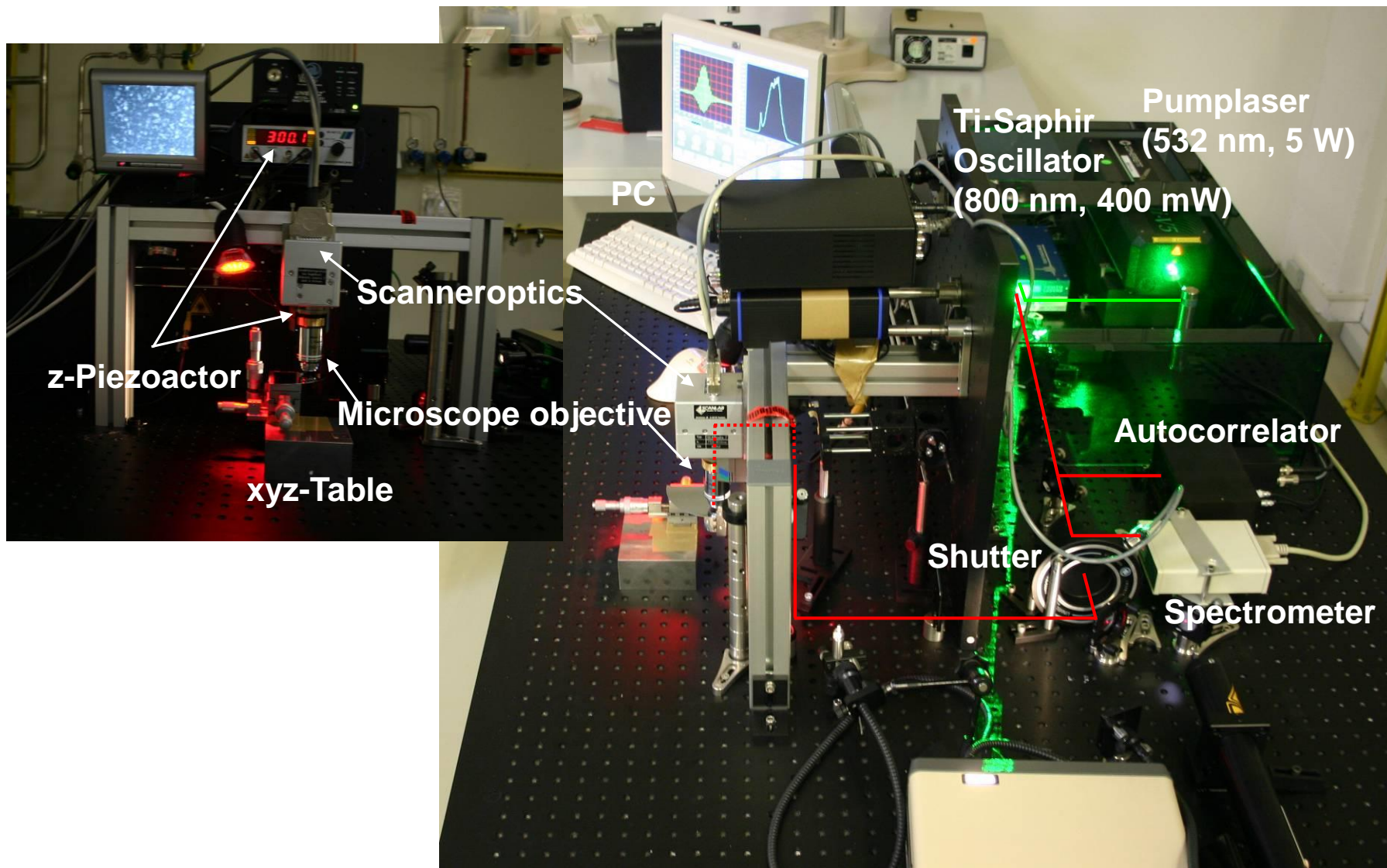


Two-Photon Microfabrication

- a single intuitive step
- 3-D structures
- spatial resolution of $< 200 \text{ nm}$



Direct Laser Writing: Experimental Setup

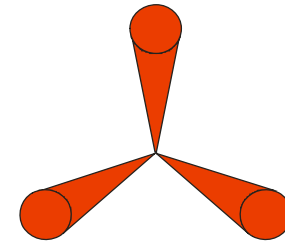
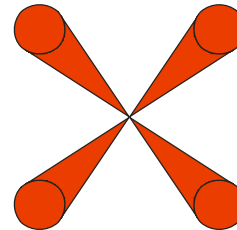
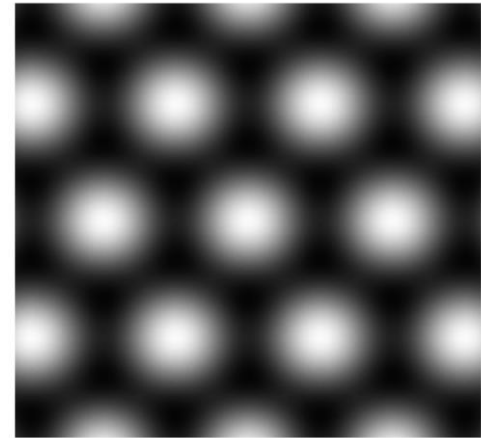
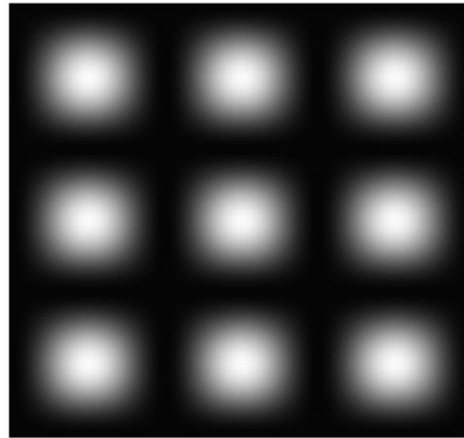
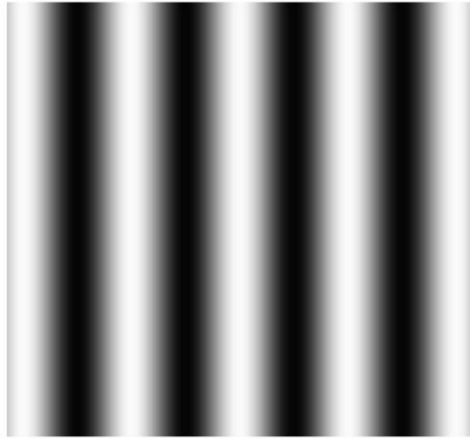


Interference Lithography: Working Principle

If two (or more) coherent laser beams overlap in space, an interference pattern with distinct bright and dark regions is generated.

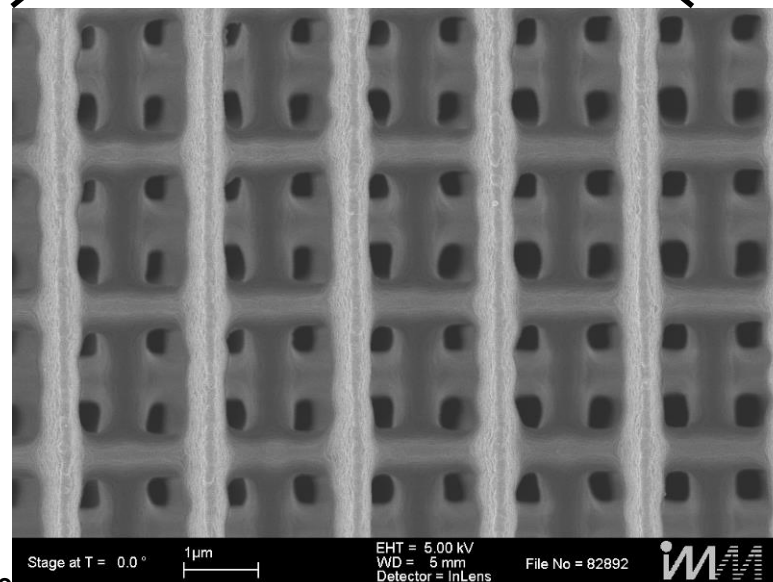
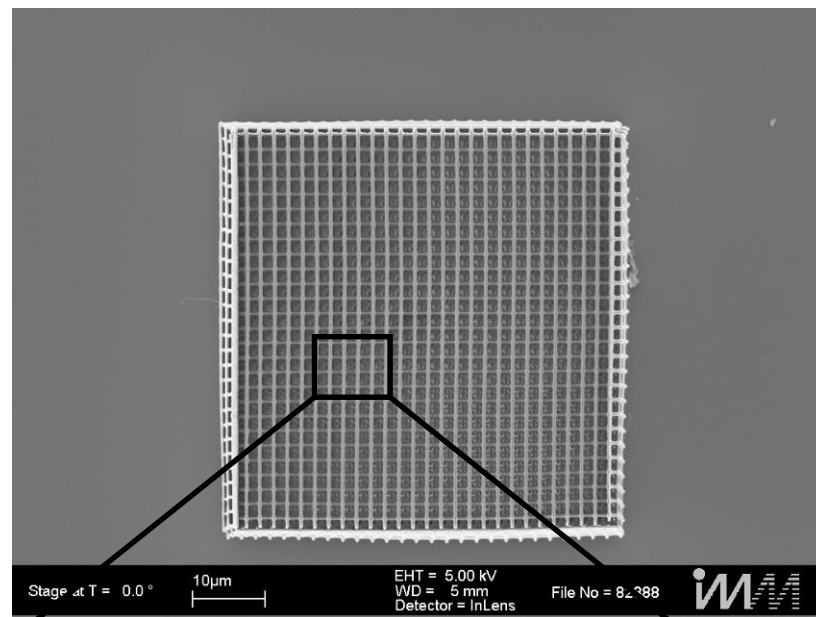
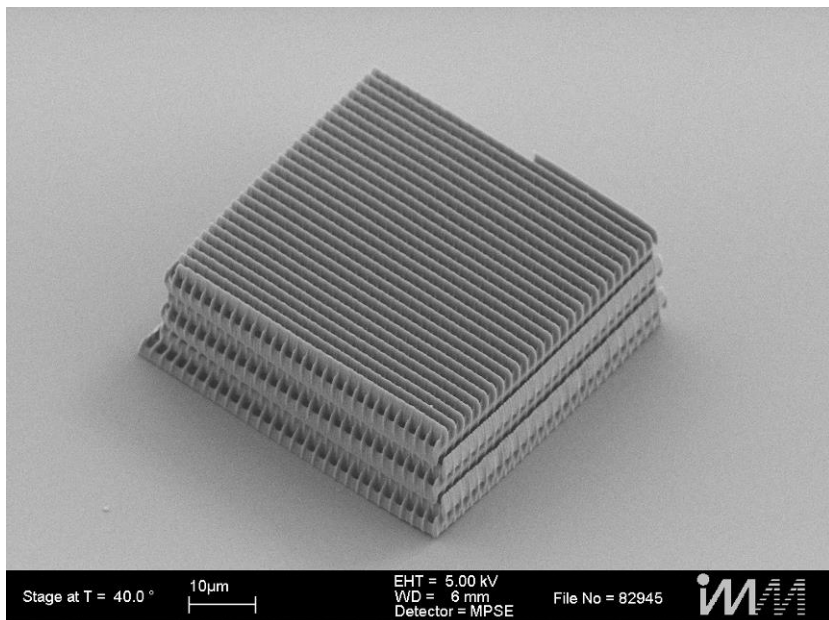
Idea: Expose a photosensitive polymer layer (photoresist) with this pattern.

Interference Lithography: Simulation



The pattern geometry is determined by the number of laser beams and their respective intensity, orientation, and polarization.

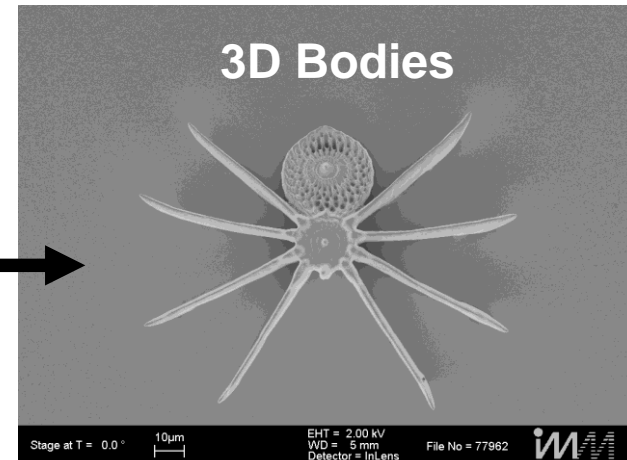
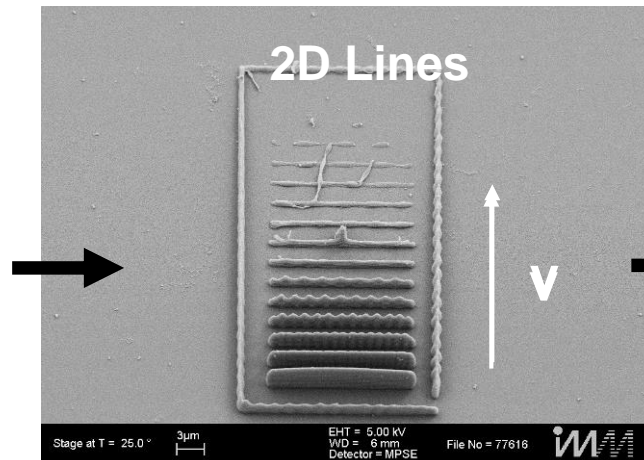
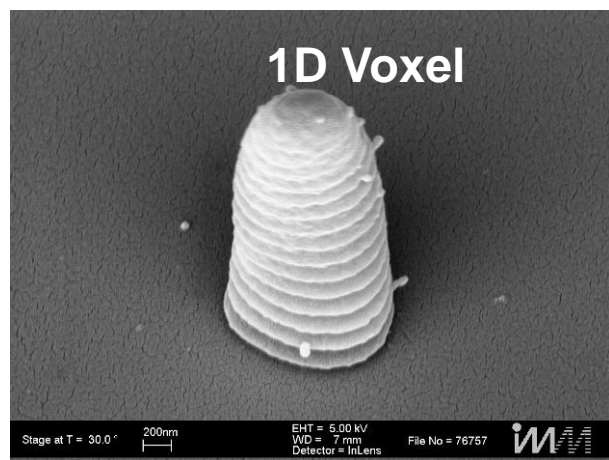
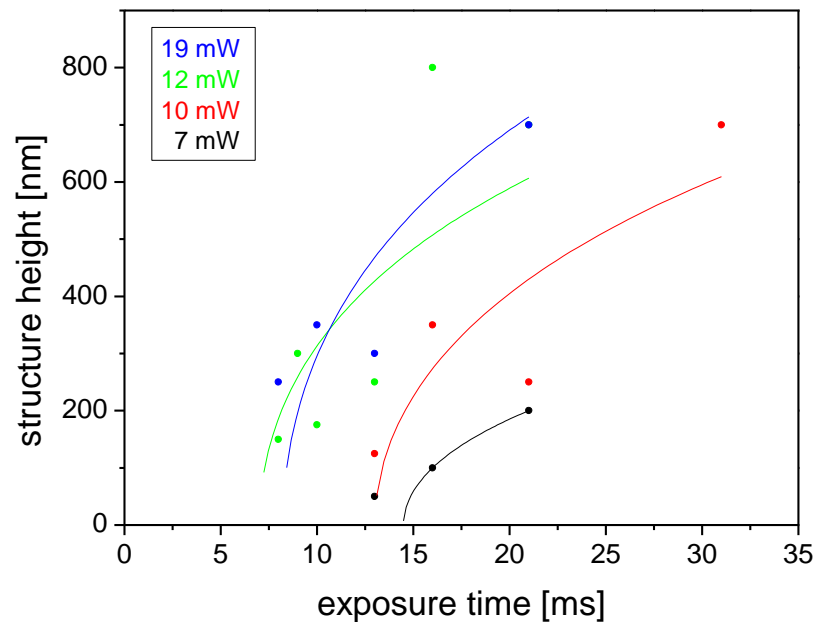
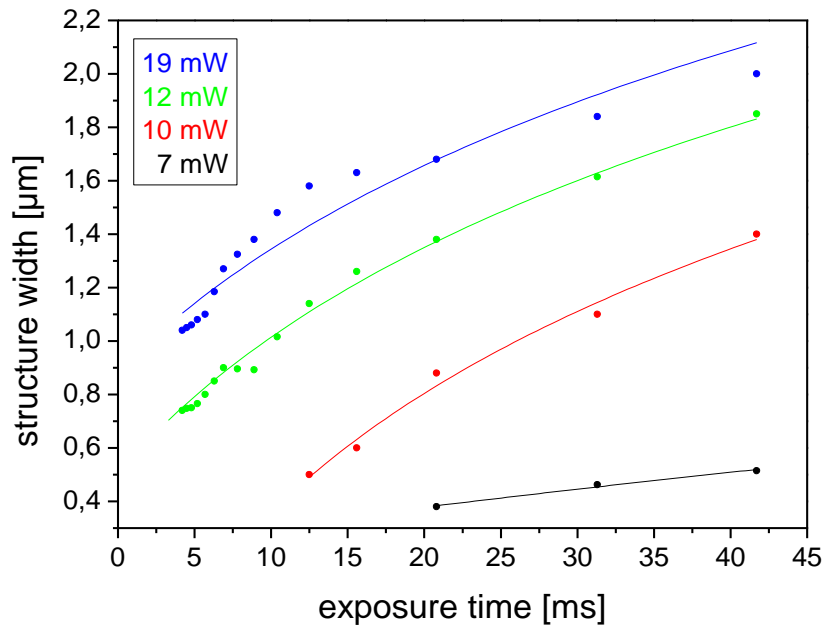
Direct Laser Writing: Wood-Pile Structures



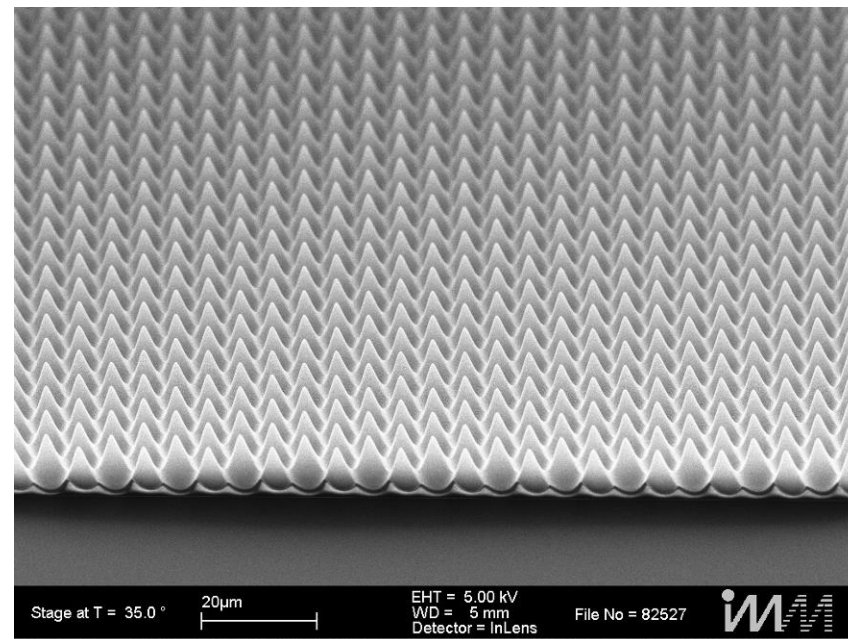
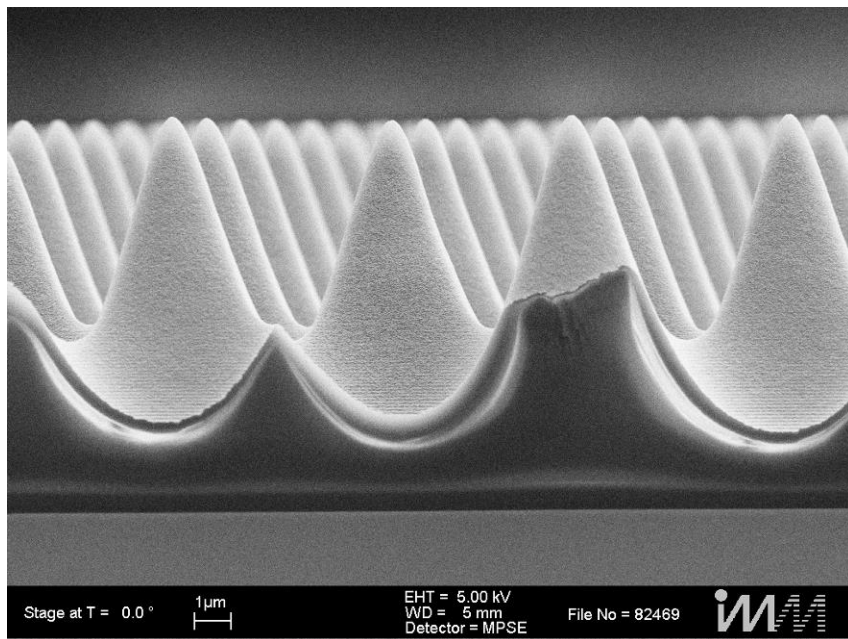
Resist: SU-8

Application: Photonic Crystals
with complete band-gap

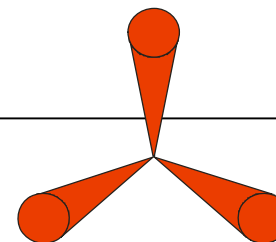
Direct Laser Writing: Results



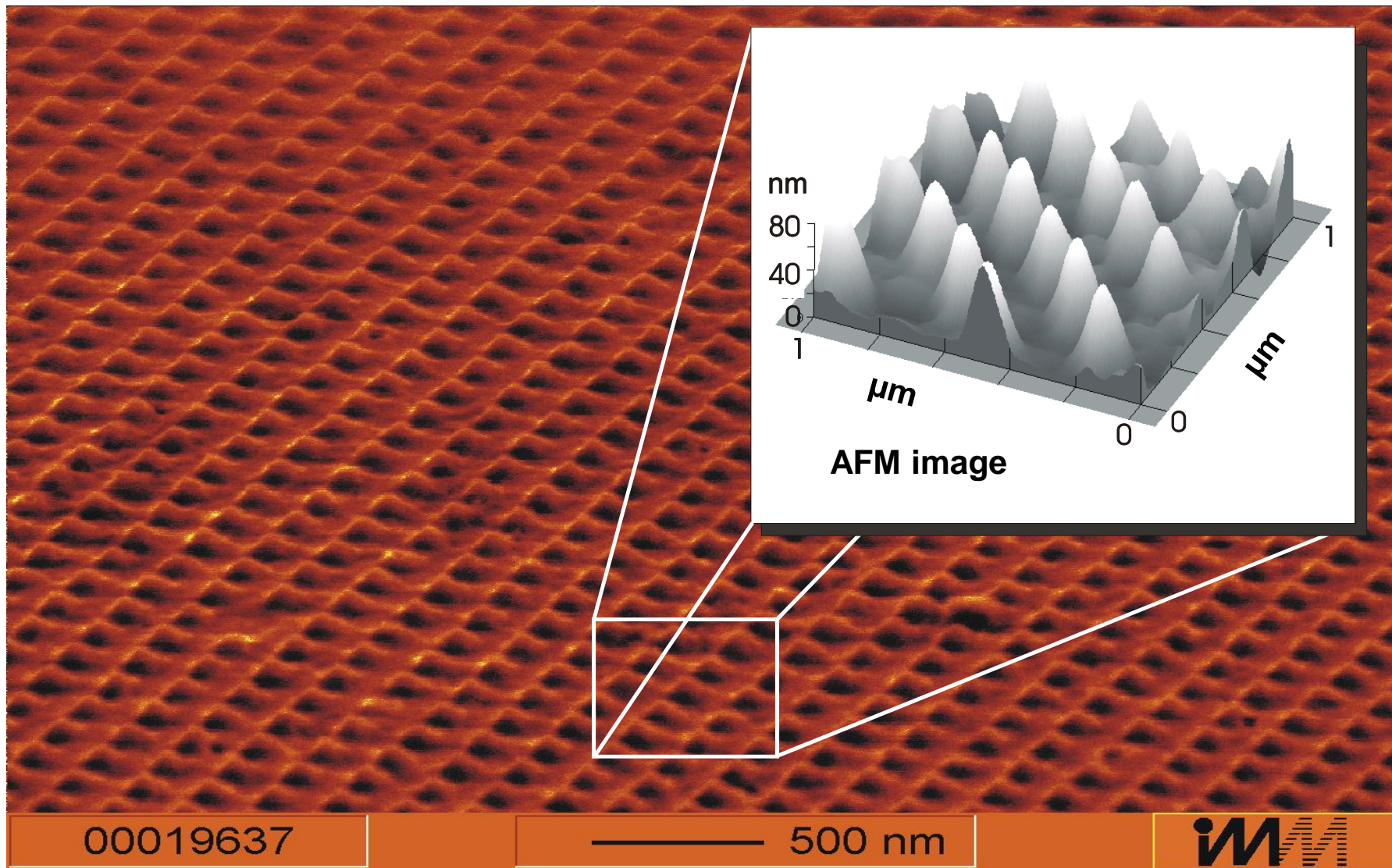
Interference Lithography: 2-D Structures



The actual pattern obtained also depends on the properties of the photoresist material.



Electroforming of Nano-tip Arrays with Nickel



00019637

500 nm

Bulk Material Micro-structuring and Surface Modifications (V)

Micro-structuring

mechanical

electrical (EDM)

chemical

irradiation

electrochemical

Surface modification

electrodeposition

PVD/CVD

polymer coatings

chemical

SAMs

Materials

metals

ceramics

glass

semiconductors

polymers

Electrodeposition

General

Gold

Copper

Nickel

Nickel-cobalt

Nickel- iron

Mold inserts

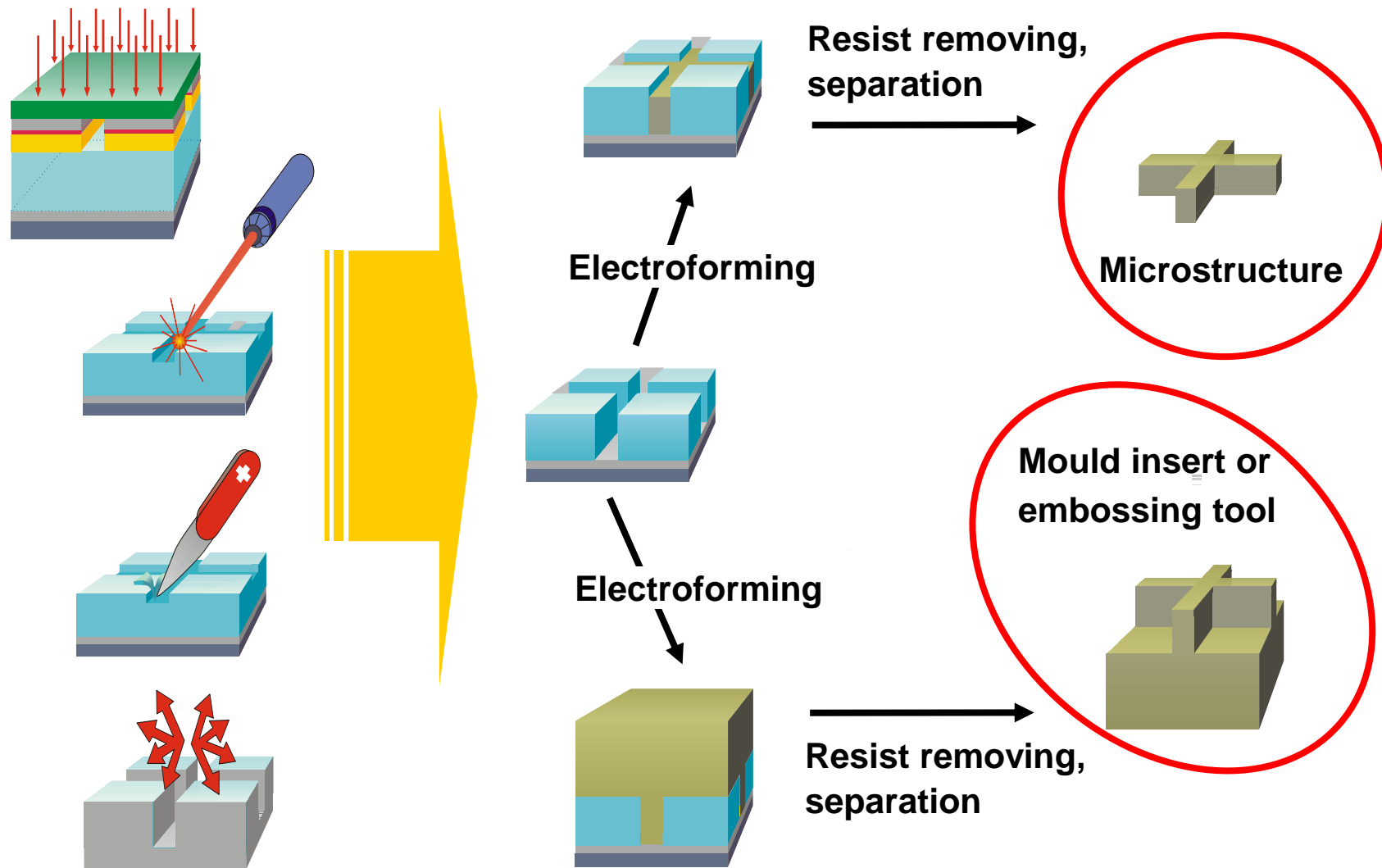
Special types of nickel alloys

Mold inserts from (ASE) silicon masters

Resist

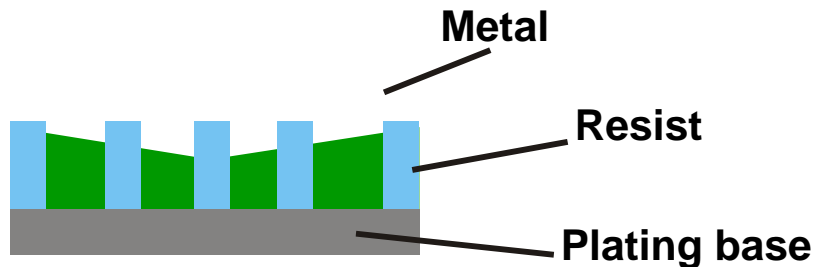
Plating equipment

Principles of Micro Machining Steps

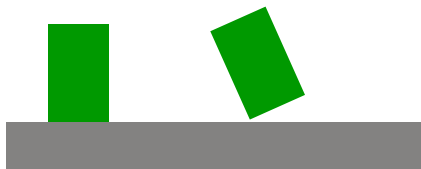


Main Problems in Microelectroforming

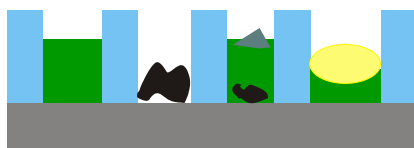
1. Metal distribution



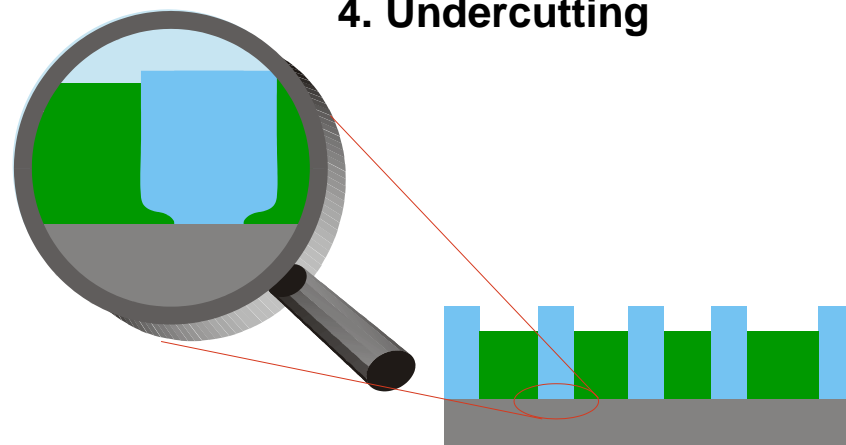
2. Adhesion



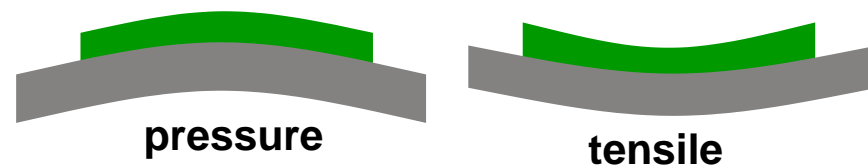
3. Particels and bubbles



4. Undercutting

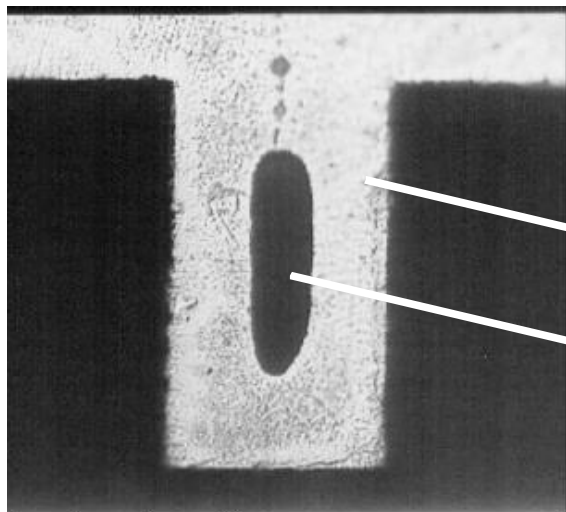


5. Internal stress





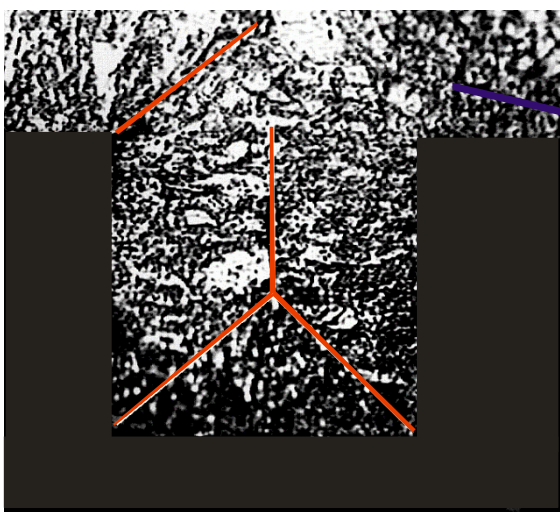
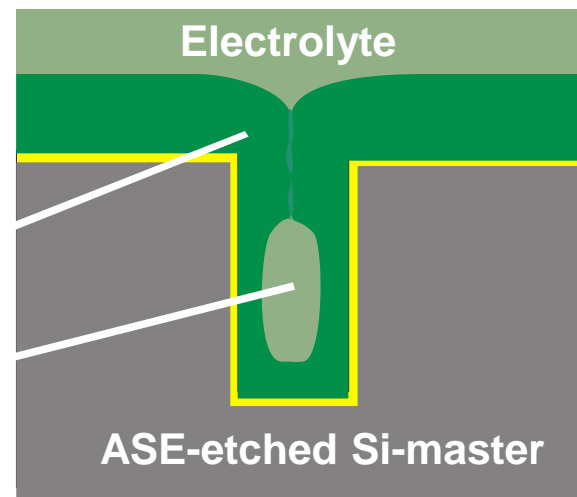
Typical Defects Caused by Electroplating of fully metalized ASE-etched Si-surfaces



Sputtered adhesion
and seed layer
Deposited nickel

Electrolyte entrapment

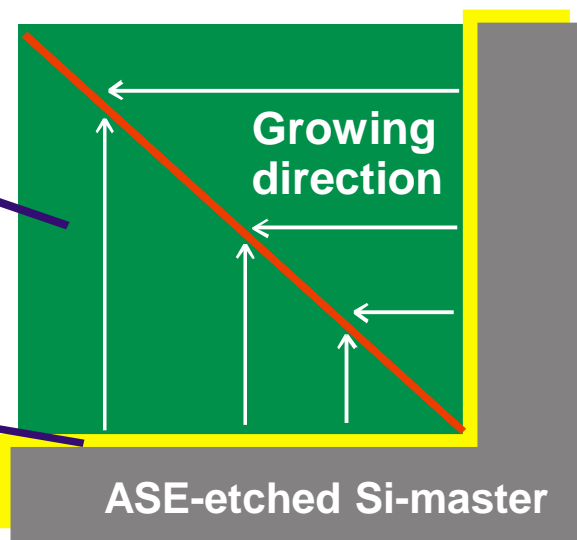
Aspect ratio 2:1



Deposited nickel

Sputtered adhesion
and seed layer

Aspect ratio 1:1



Electrodeposition

General

Gold

Copper

Nickel

Nickel-cobalt

Nickel- iron

Mold inserts

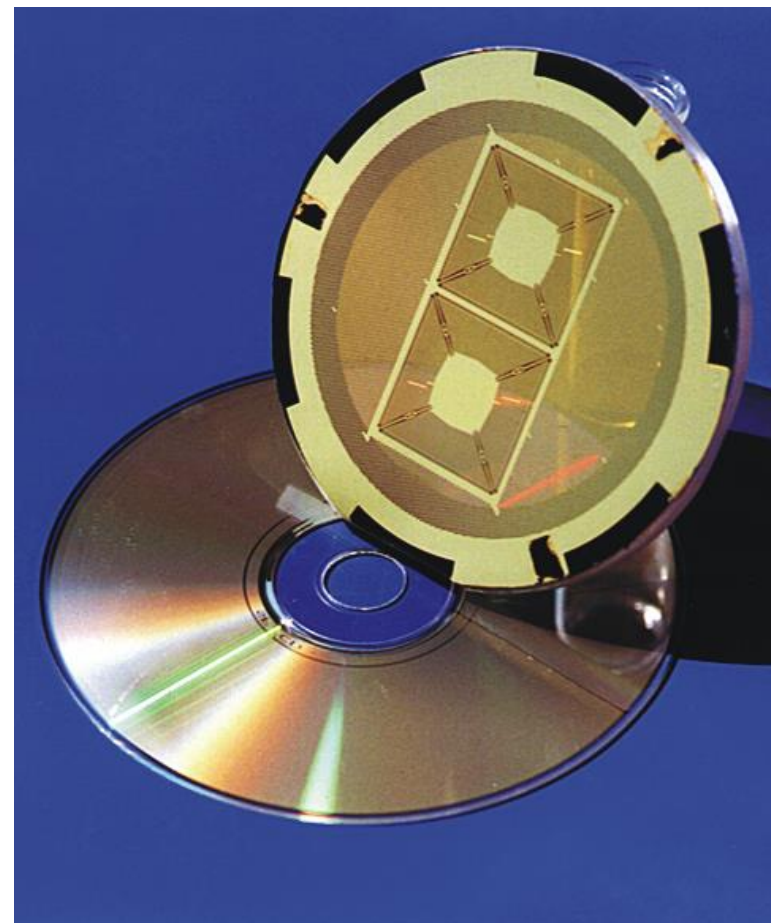
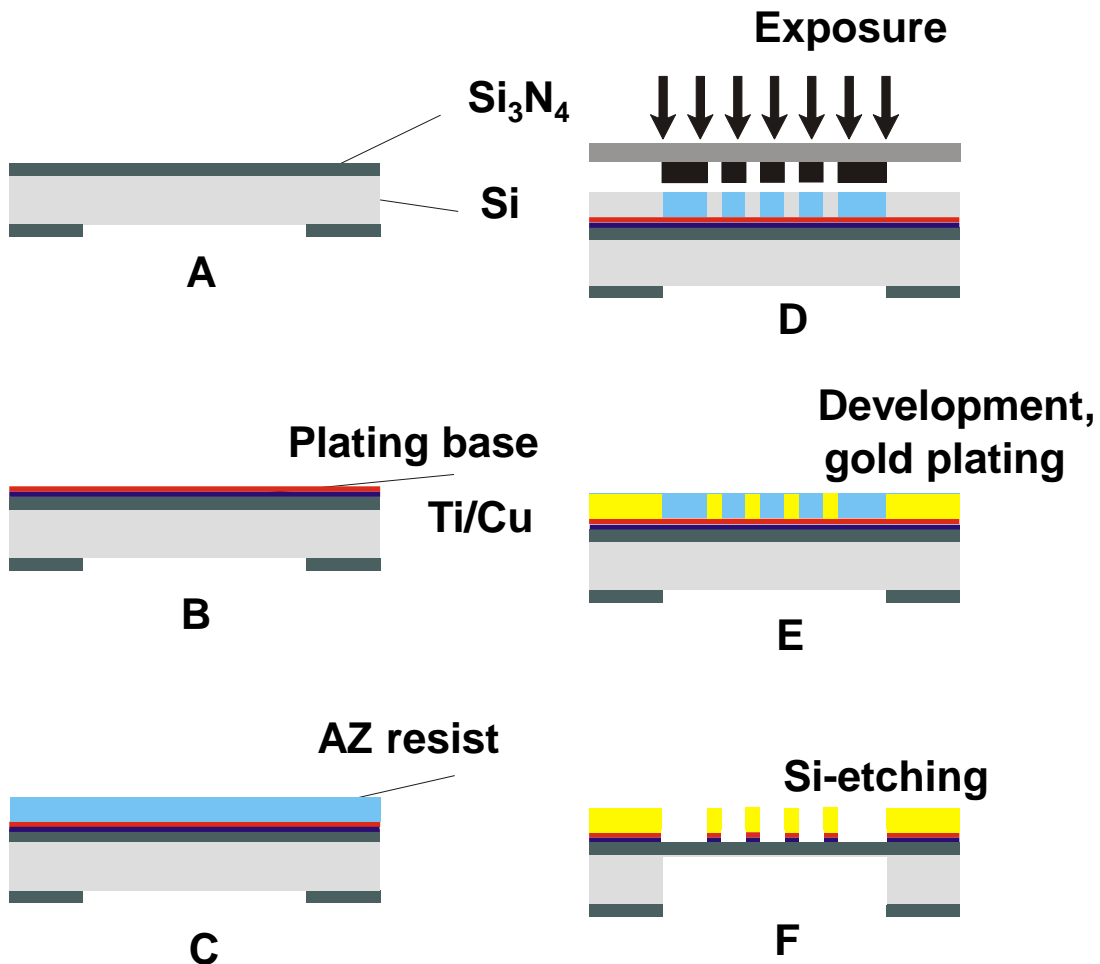
Special types of nickel alloys

Mold inserts from (ASE) silicon masters

Resist

Plating equipment

X-ray Mask Plating Process



2µm Au-pattern

Electrodeposition of Gold: Working Conditions

Gold electrolyte

**Modified commercial available
gold sulfite electrolyte**

Gold content

8 - 12 g/l

Sulfite as sodium sulfite

40 g/l

Leveler

Arsenite / arsenate

pH - value

9.3 - 9.6

Temperature

55° C

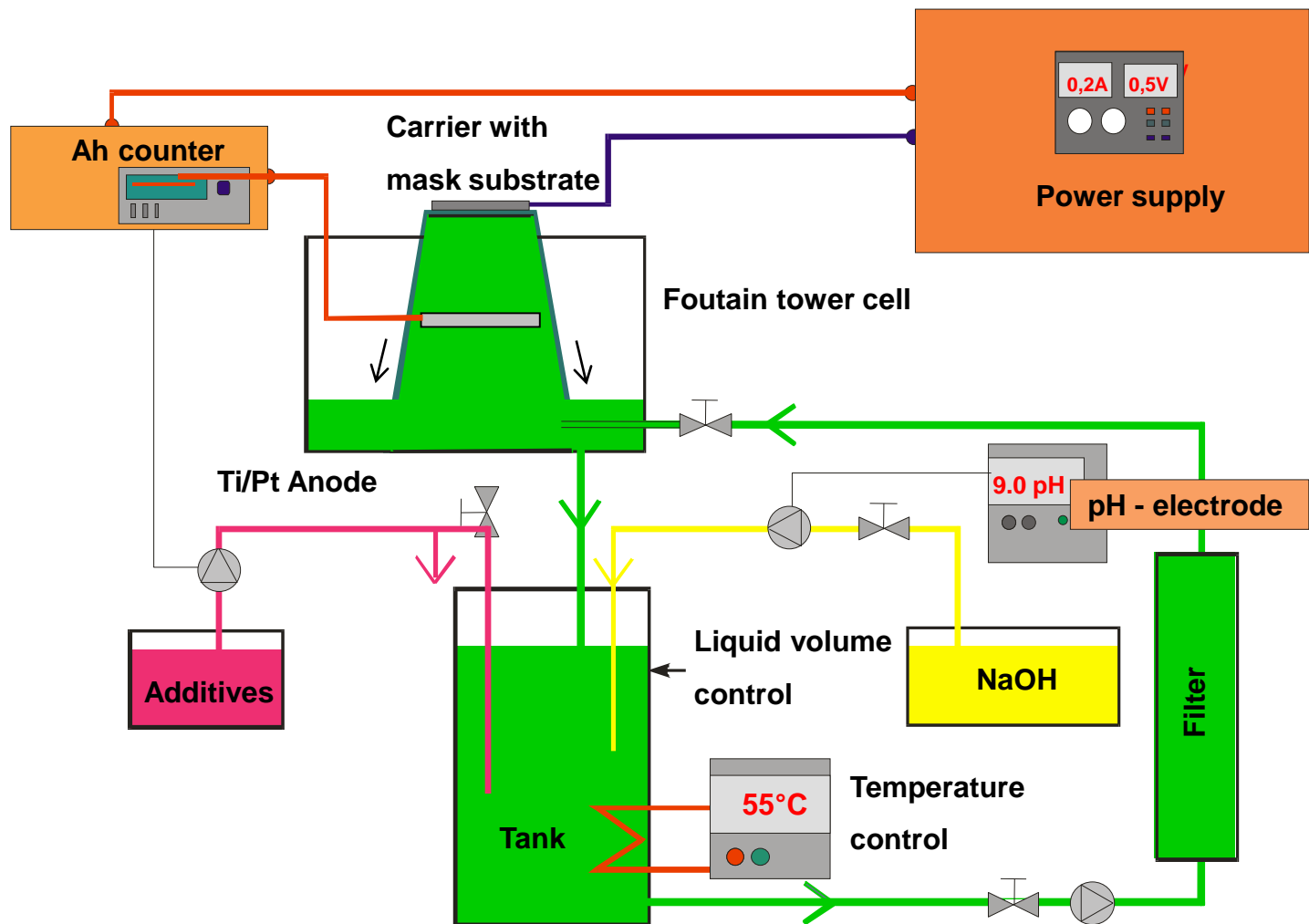
Current density

0.2 - 0.4 A/dm²

Anode material

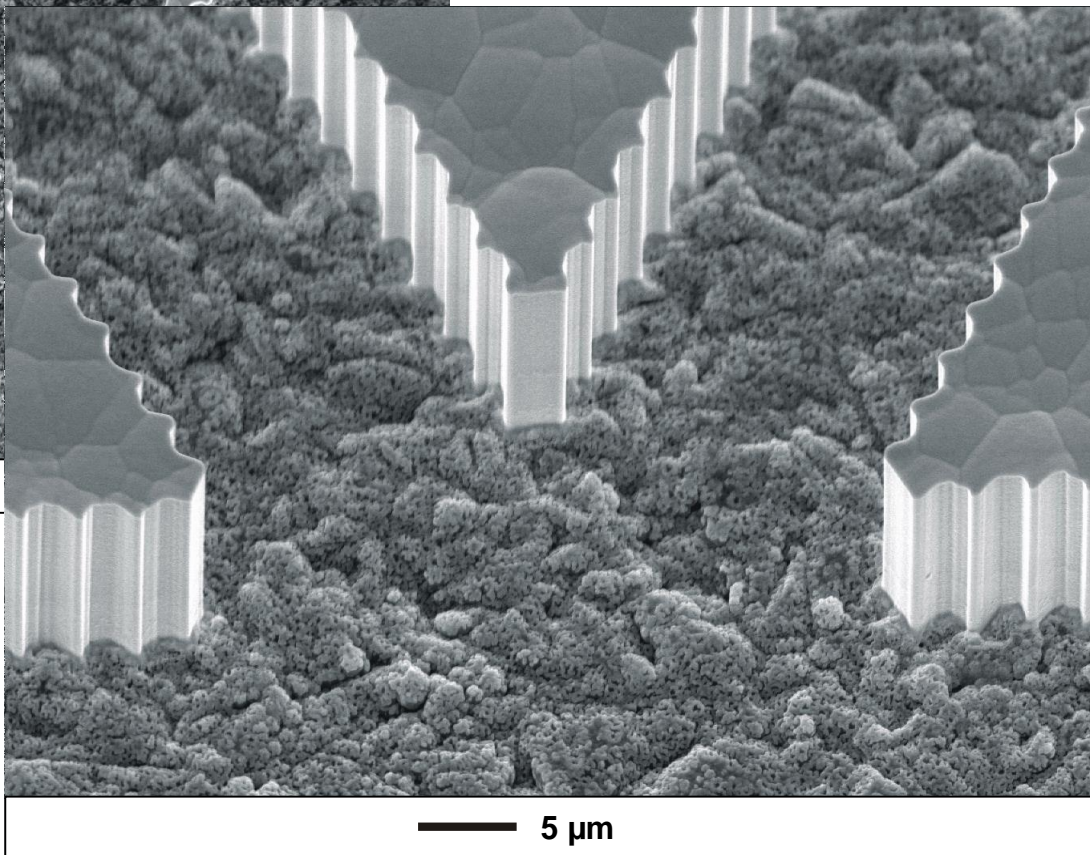
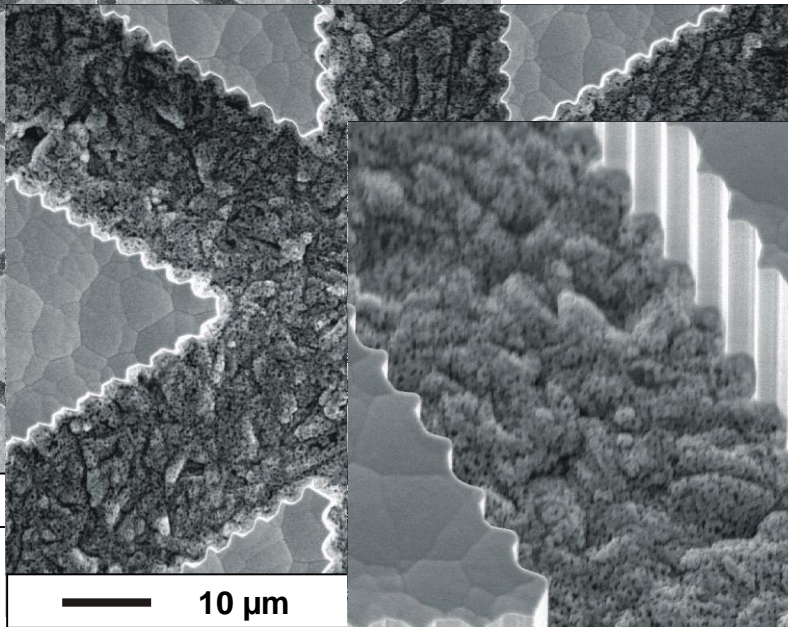
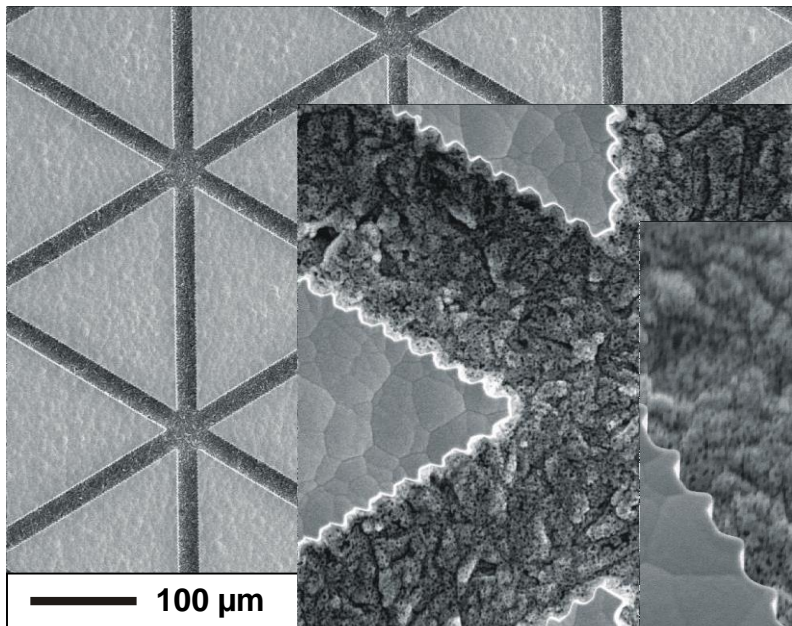
Ti/Pt mesh

Mask Technology: Gold Electrodeposition in a Fountain Tower Cell



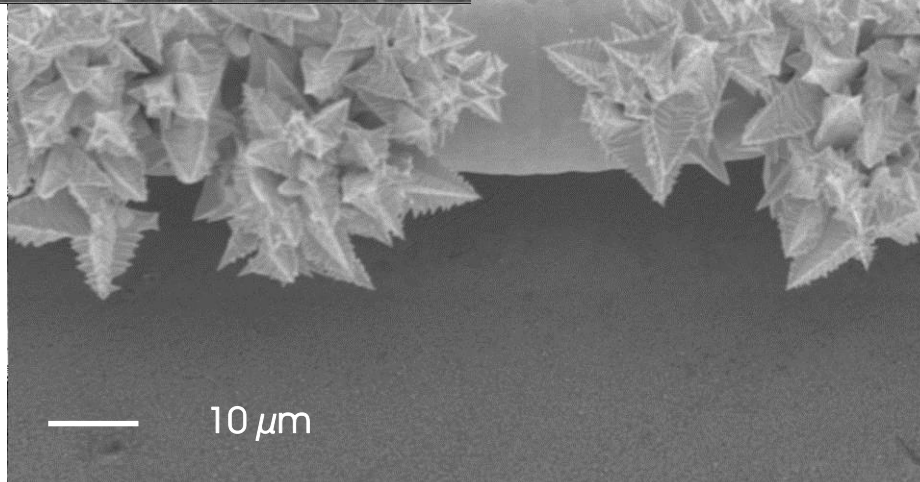
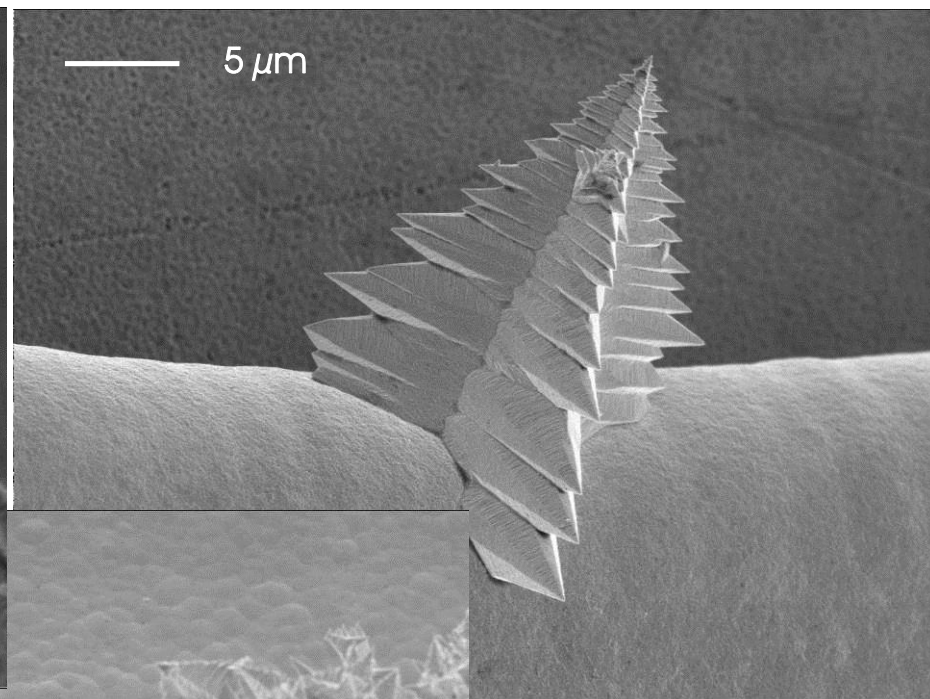
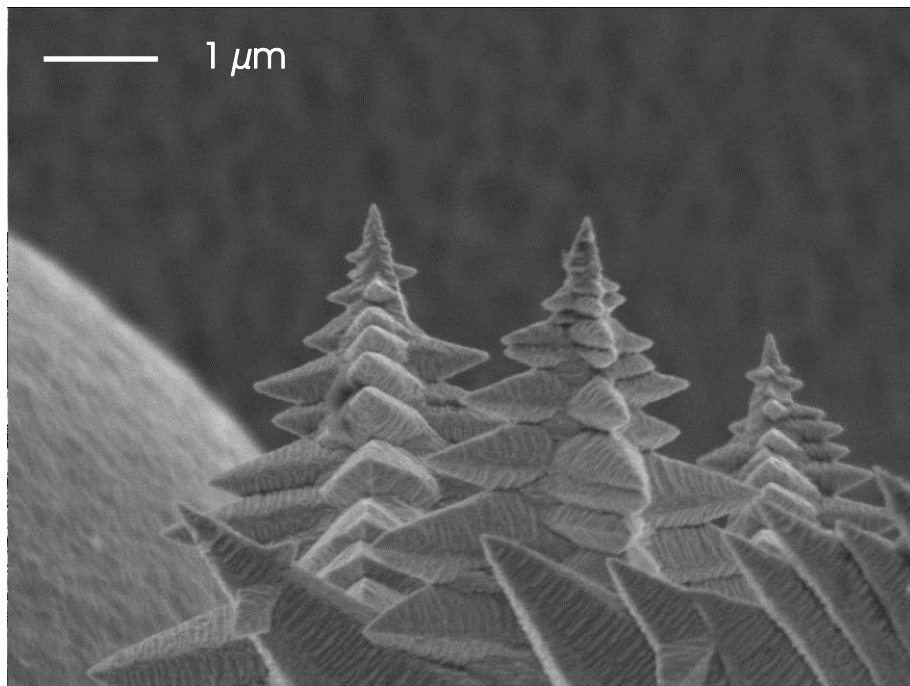


High Leveling Gold Electrodeposition

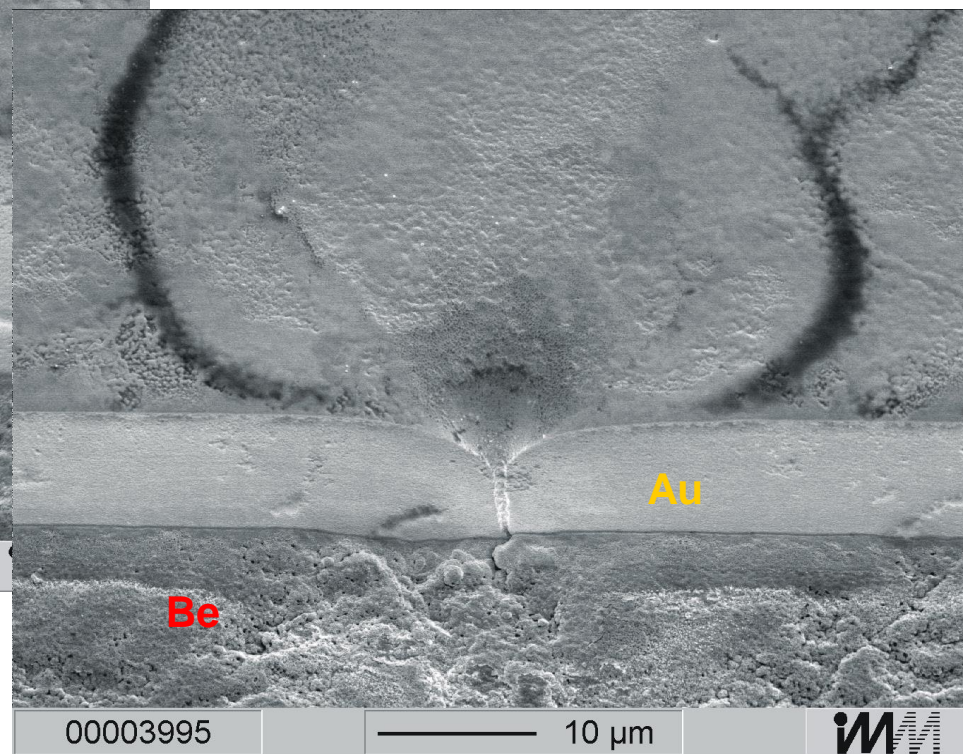
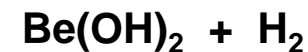
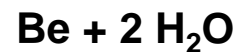
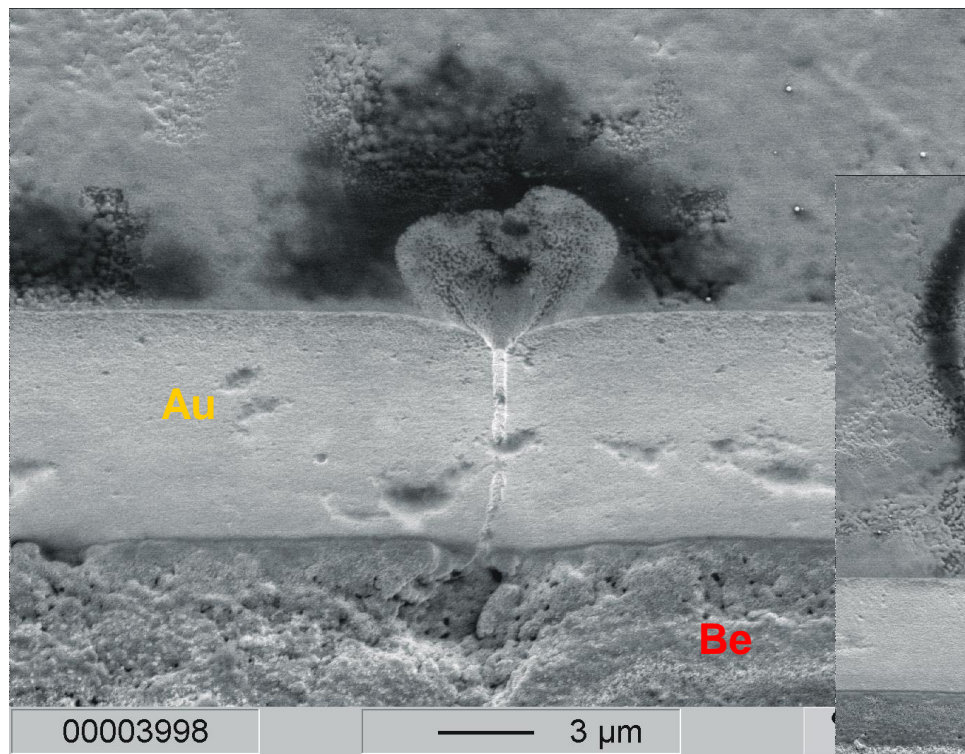


Thickness: 20 μm

Mistakes in Electroplating of Gold - Leveler Concentration Too Low

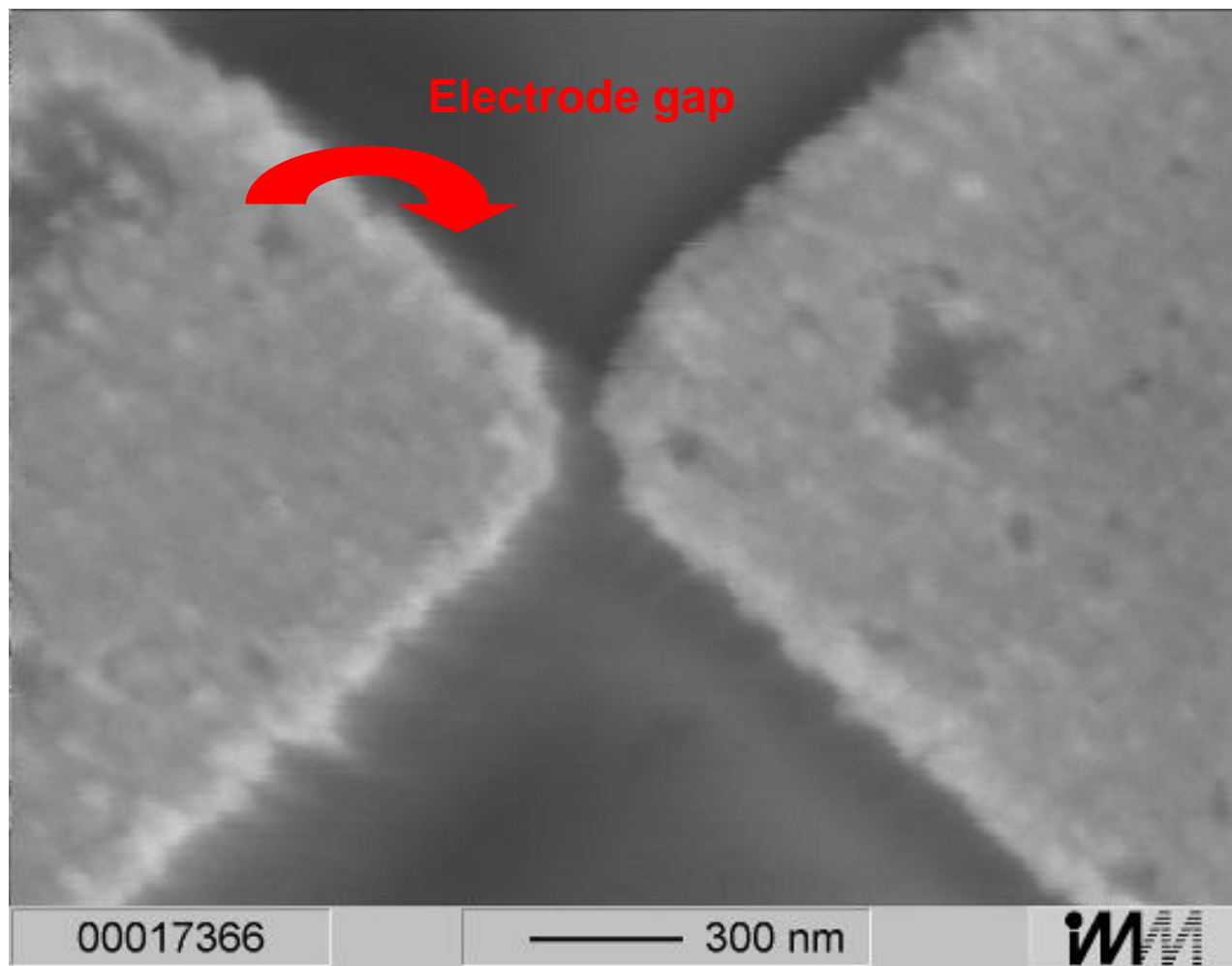


Deep X-ray Lithography: Substrate Induced Defects



Formation of gas bubbles by dissolution of the Be-substrate during the electrodeposition process

Electroplating with AFM Tips: Gold Nanoelectrodes



Electrodeposition

General

Gold

Copper

Nickel

Nickel-cobalt

Nickel- iron

Mold inserts

Special types of nickel alloys

Mold inserts from (ASE) silicon masters

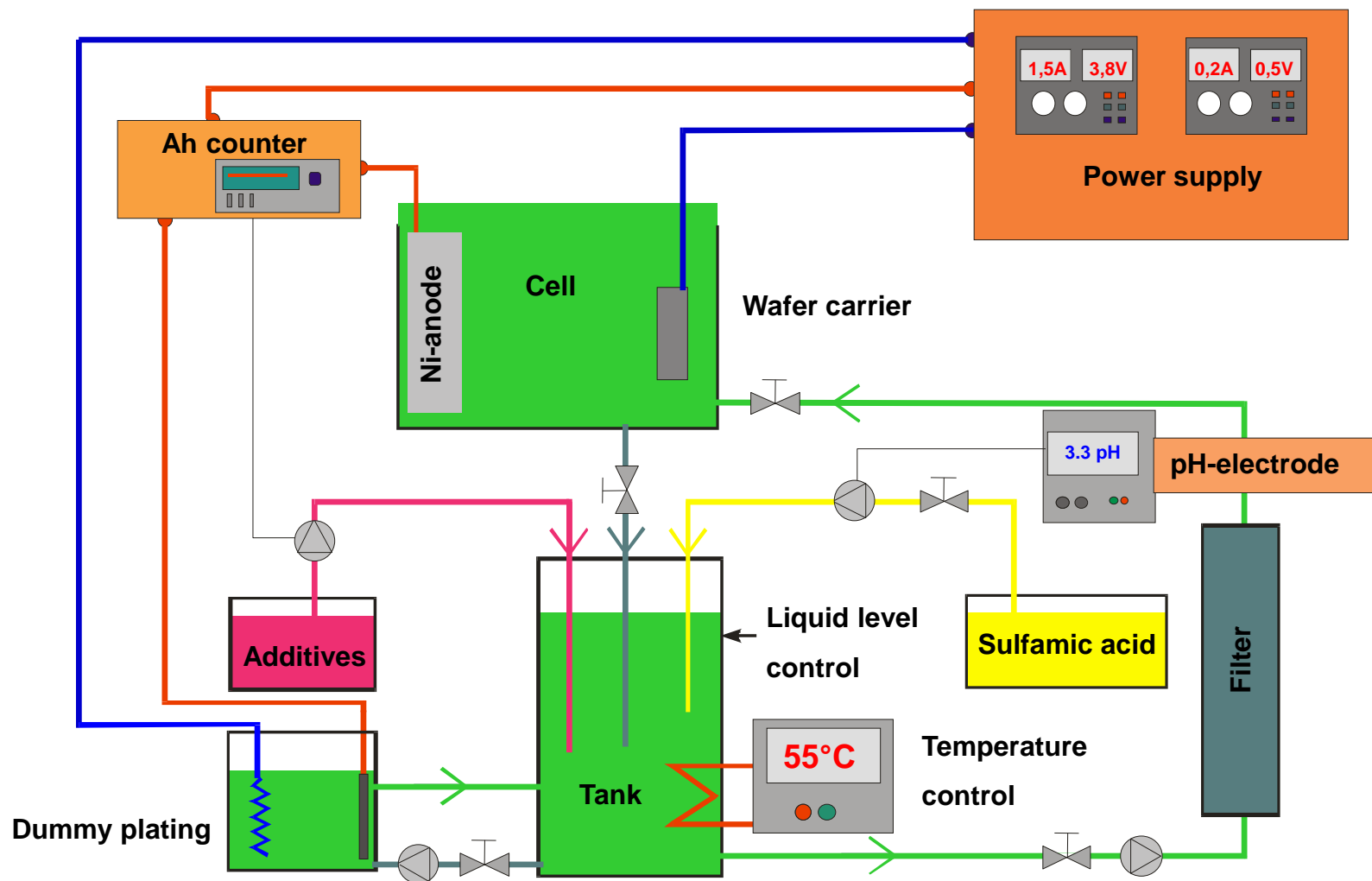
Resist

Plating equipment

Nickel Sulfate Electrolyte: Working Conditions

Nickel	100 g/l	Anode reaction:
Boric acid	40 g/l	$\text{Ni}^{\pm 0} \rightleftharpoons \text{Ni}^{2+} + 2e$
Sulfaminic acid	275 g/l	
Sulfate	14 g/l	Cathode reaction:
Chloride	5.0 g/l	$\text{Ni}^{2+} + 2e \rightleftharpoons \text{Ni}^{\pm 0}$
Saccharin	20 mg	$2\text{H}^+ + 2e \rightleftharpoons \text{H}_2$
fluorinated wetting agent	20 ml/l (2% solution)	
Anode material	Sulfur depolarized nickel	
Temperature	55 °C	
pH - value	3.8	
Current density	1 - 5 A/dm ²	
Current efficiency	~ 99%	
Bath volume	75 l	

Nickel Electrodeposition: Flow Chart



Fiber Alignment Device

Deep UV-lithography

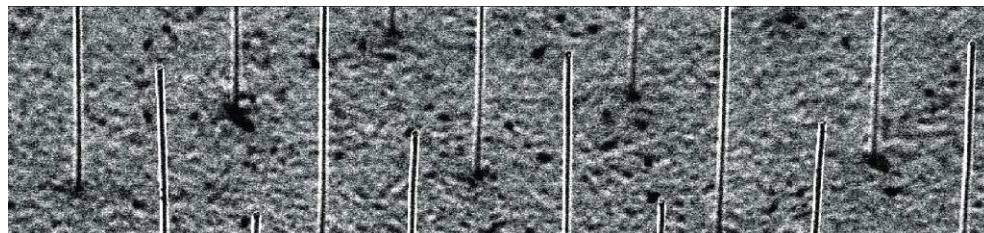
electroforming
with nickel
Sacrificial layer
technique

00005346

— 100 μm

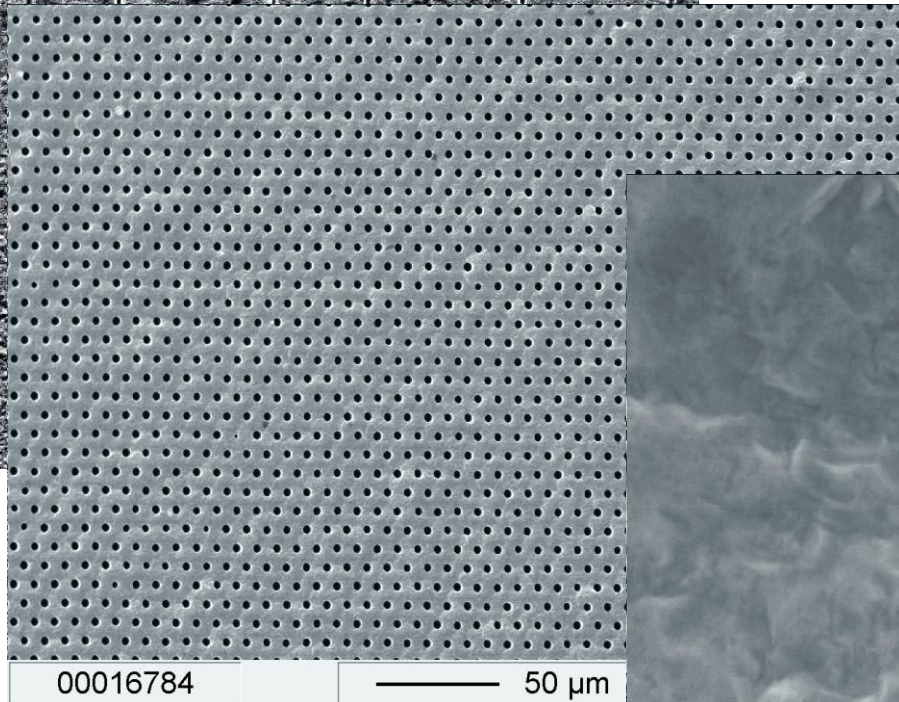


Negative Tone - Resist for Deep X-ray Lithography: Columns Replicated by Electroforming



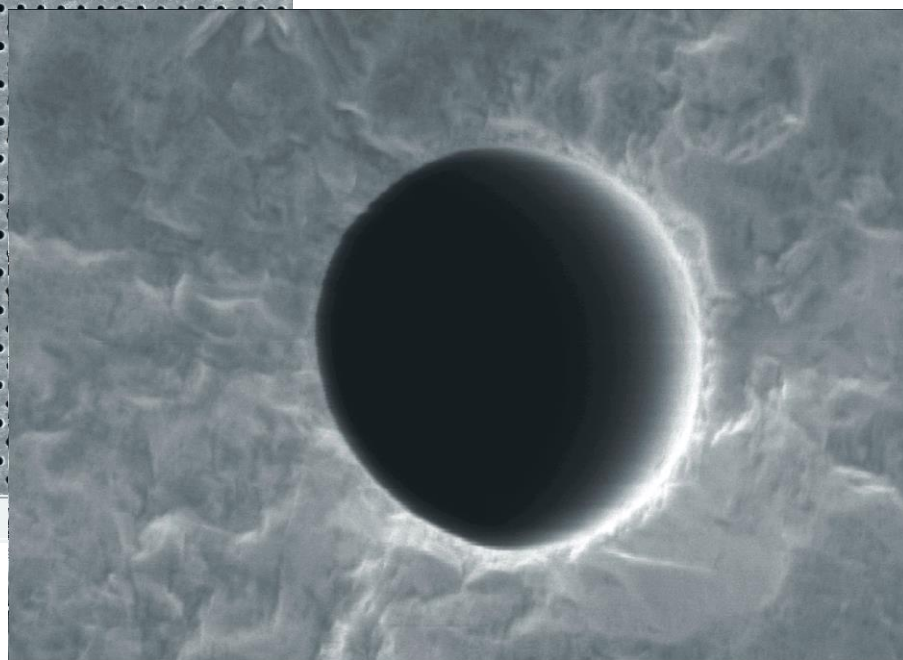
Columns:
Diameter: 3 μm
Height: 50 μm

Material: Nickel
Height: 45 μm
Surface not polished



00016784

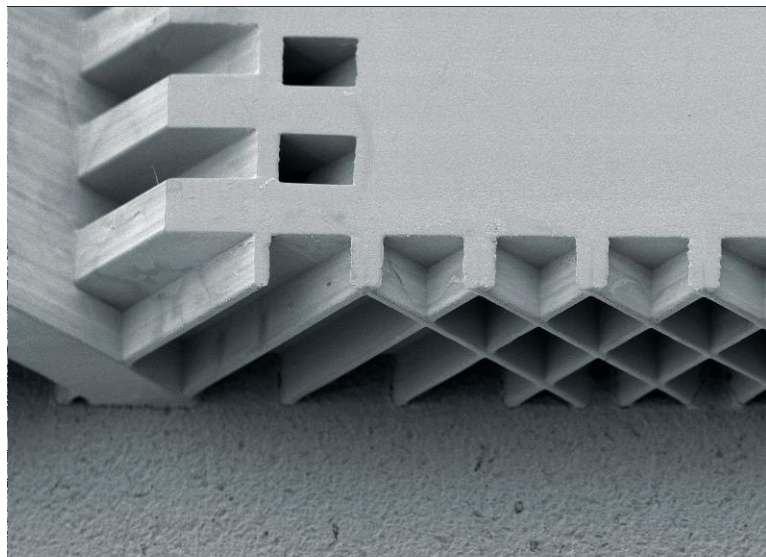
50 μm



00016783

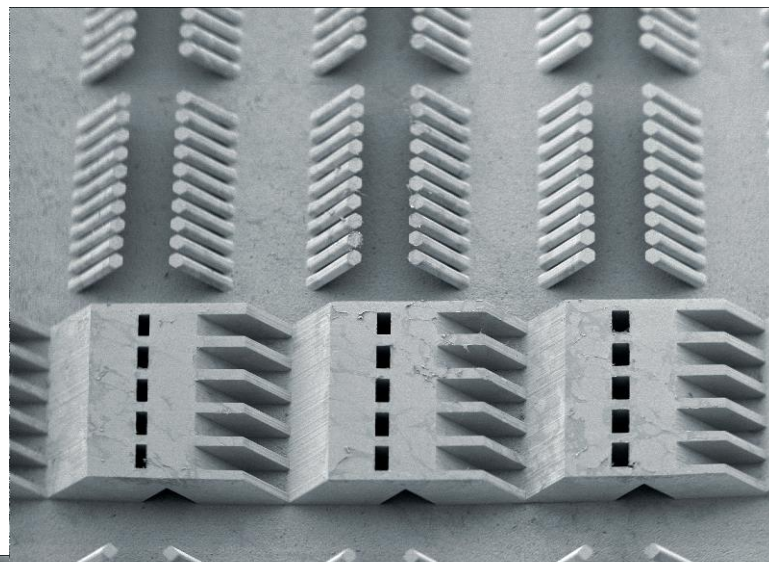
1 μm

LIGA - Microstructures Made from Nickel: Oblique X-ray Irradiation



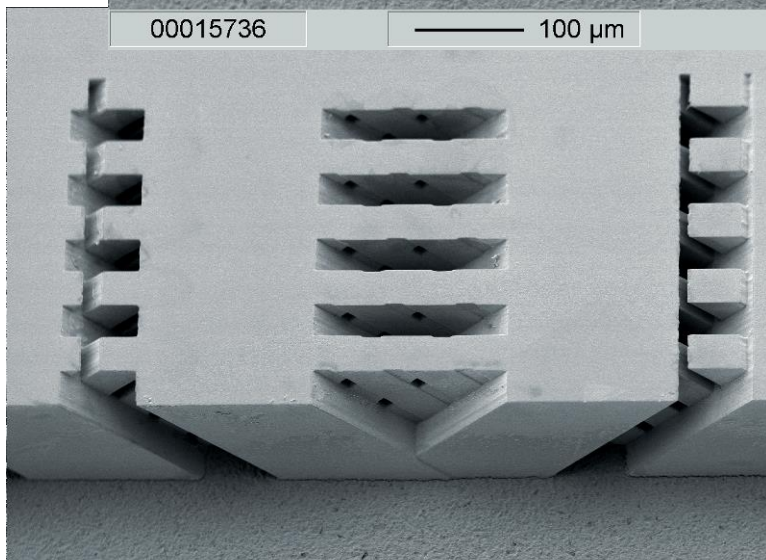
00015736

— 100 μ m



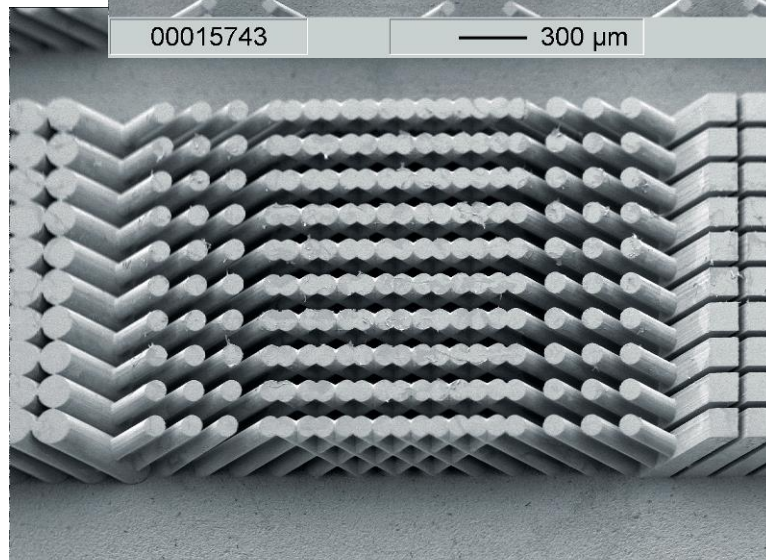
00015743

— 300 μ m



00015741

— 100 μ m



00015742

— 100 μ m

3-D Metallic Microstructures

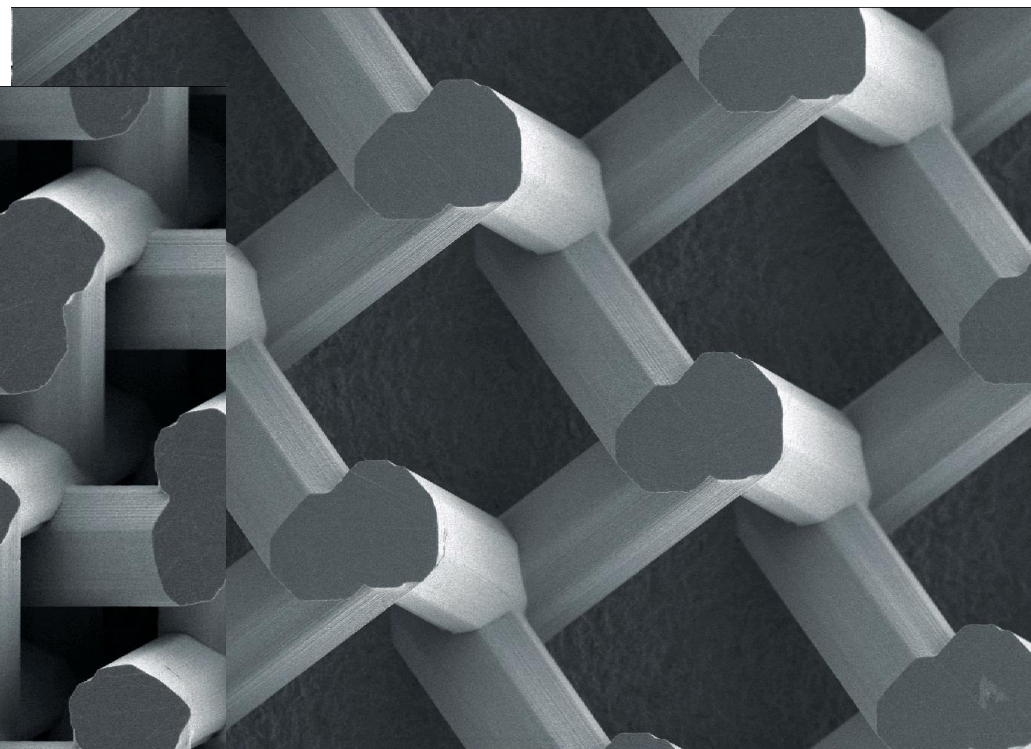
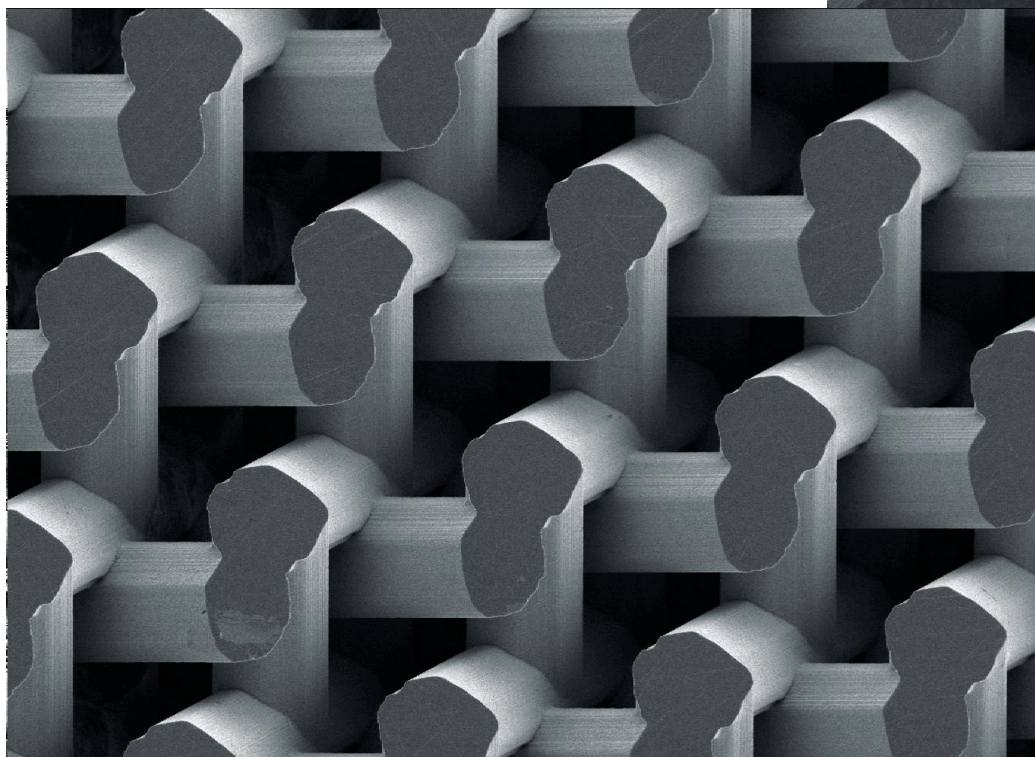
Technology:

LiGA technique, multiple oblique irradiation with rotation of mask and substrate by angular steps of 120°

Application:

Photonic crystals

Material: nickel



00025286

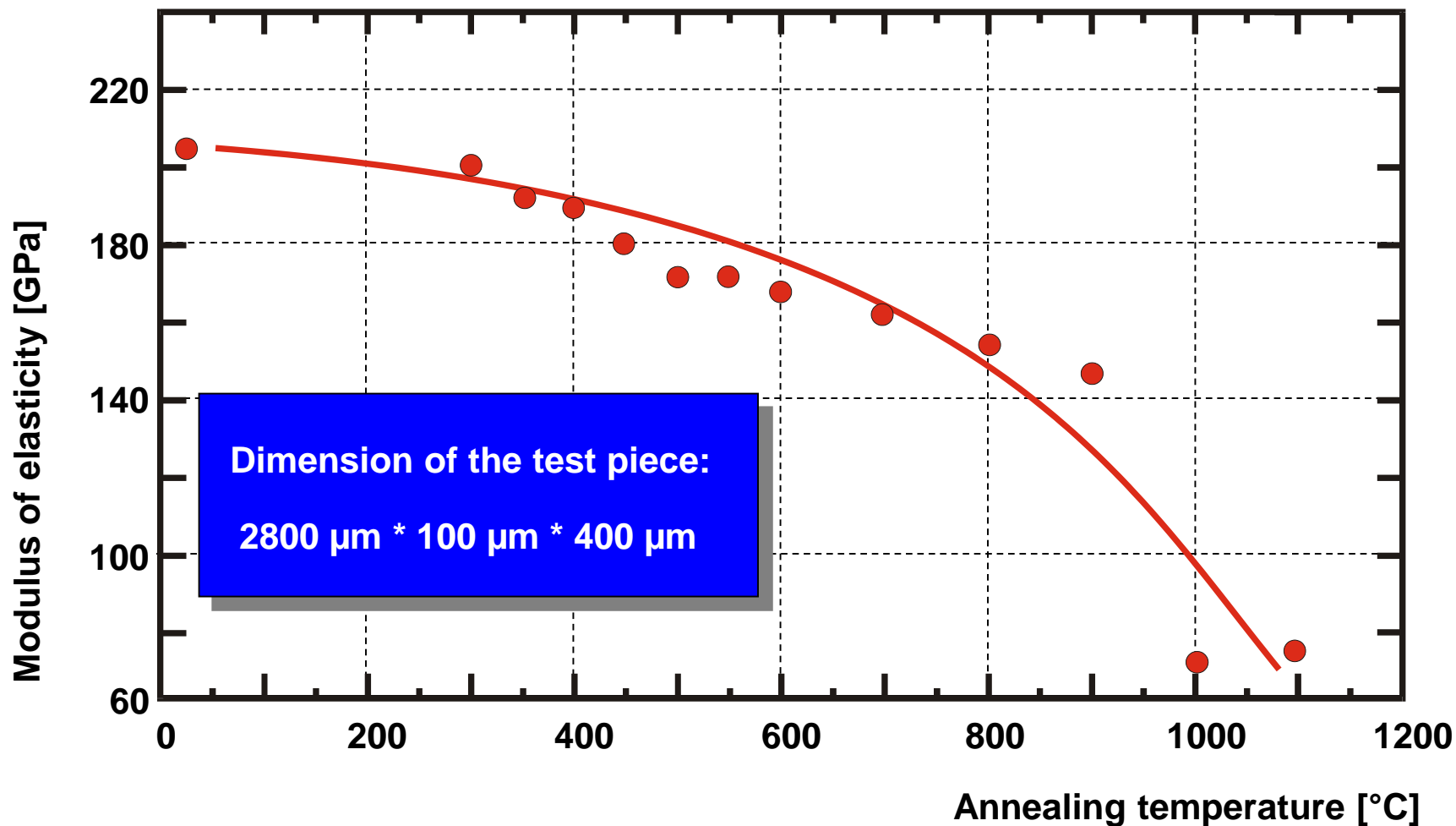
— 30 μm



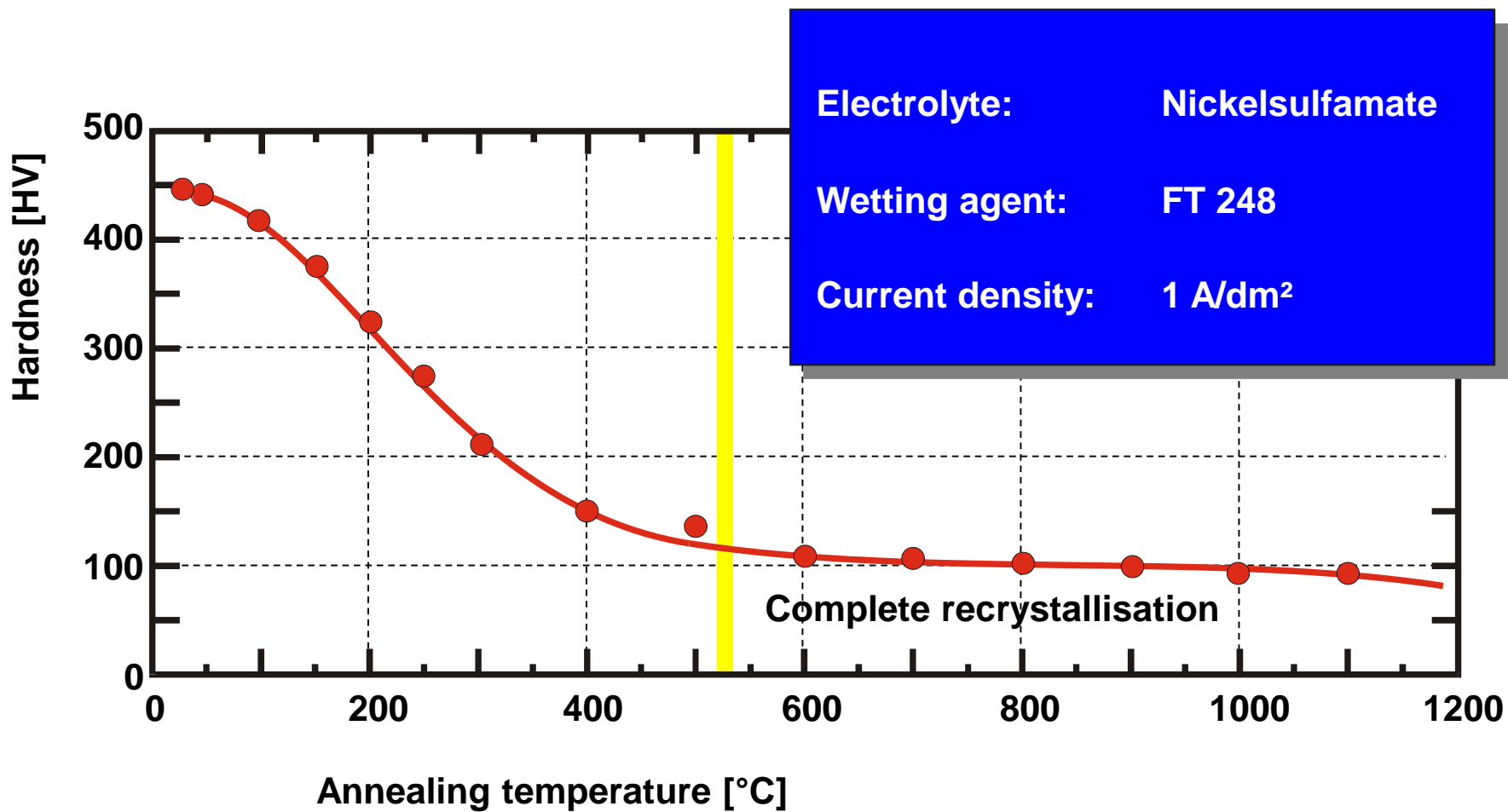
— 50 μm



Young's Modulus of Nickel Microstructures



Hardness of Ni-microstructures vs. Annealing Temperature



Electrodeposition

General

Gold

Copper

Nickel

Nickel-cobalt

Nickel- iron

Mold inserts

Special types of nickel alloys

Mold inserts from (ASE) silicon masters

Resist

Plating equipment

Nickel-Cobalt Sulfate Electrolyte: Working Conditions

Nickel	90 g/l
Cobalt	2.8 g/l
Boric acid	40 g/l
Sulfamic acid	200 g/l
Sulfate	15 g/l
Chloride	1.8 g/l
Saccharin	20 mg
fluoriertes Wetting agent	20 ml/l (2% solution)

Temperature 55 °C

pH - value 4.0

Current density 1 - 5 A/dm²

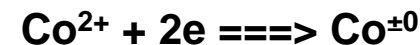
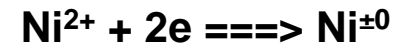
Current efficiency ~ 97%

Bath volume 75 l

Anode reactions:



Cathode reactions:



Nickel-cobalt Sulfamate Electrolyte: Mass Balance

Cathode reactions:



$j =$

$$j_{\text{Ni}} + j_{\text{Co}} + j_{\text{H}}$$



$$j_{\text{Ni}} + j_{\text{Co}} + j_{\text{H}}$$

Anode reactions:



$J_{\text{Ni}} =$

$$J_{\text{Ni(Cell)}} +$$

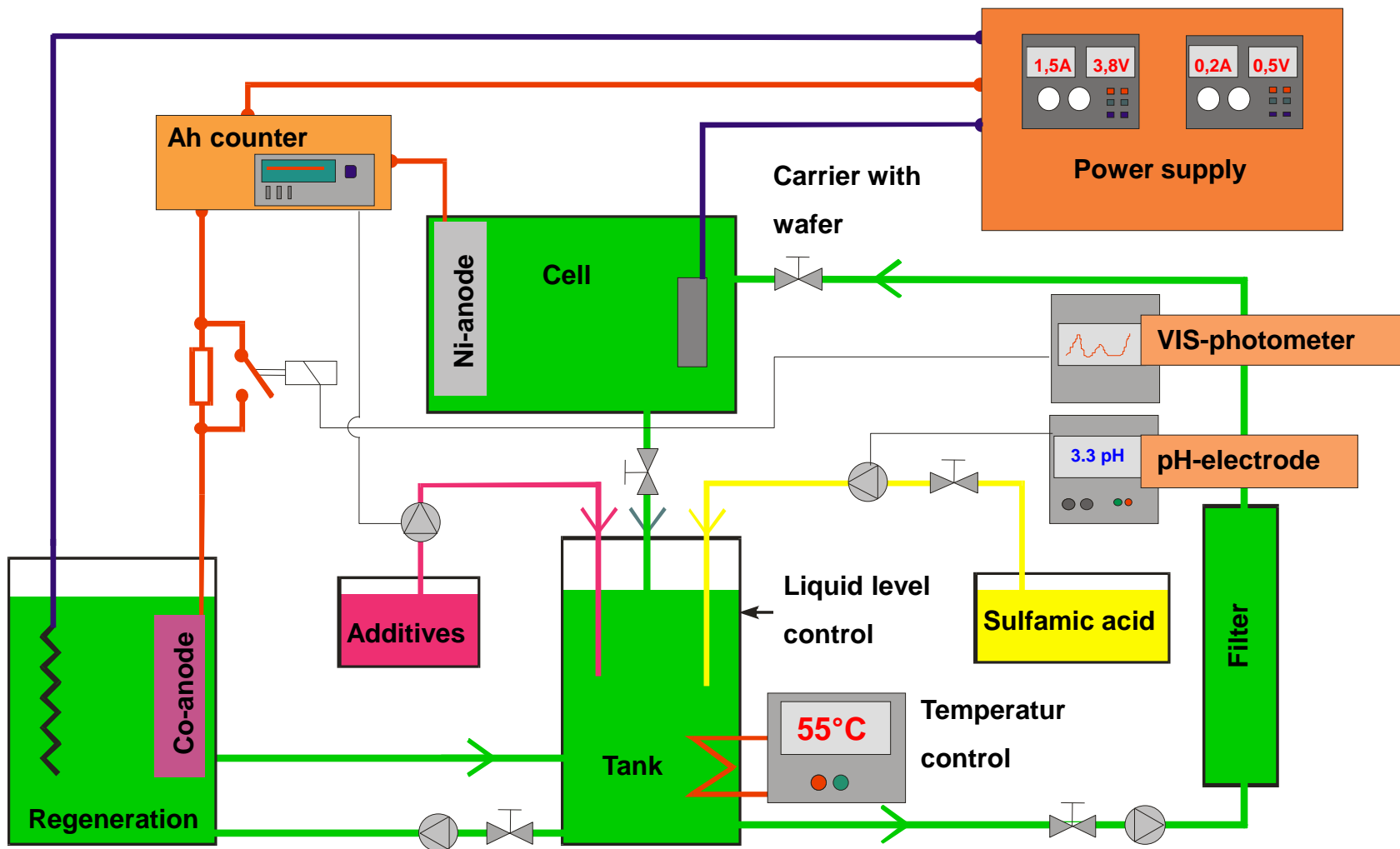
$$J_{\text{Ni(Regeneration)}}$$

$J_{\text{Co}} =$

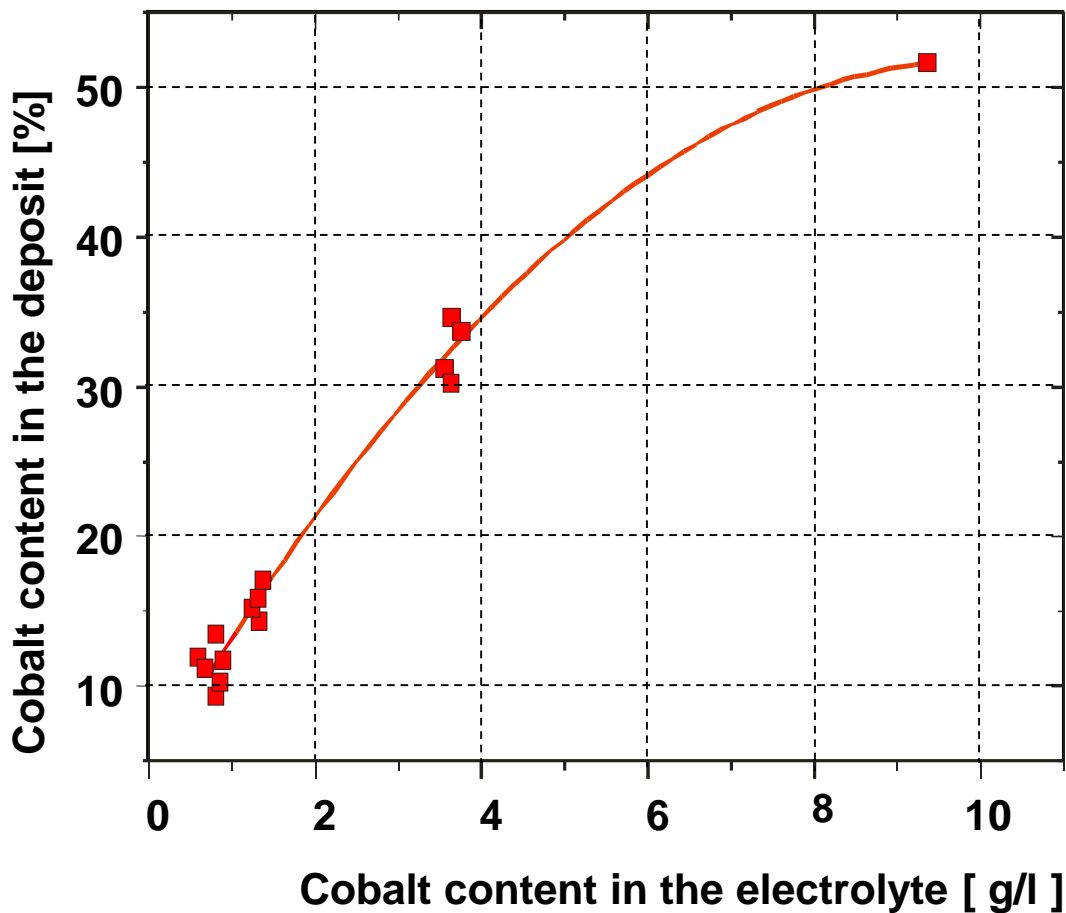
$$J_{\text{Co(Cell)}} +$$

$$J_{\text{Co(Regeneration)}}$$

Ni-Co Alloy Electrodeposition: Flow Chart



Electroplating of Ni-Co Microstructures

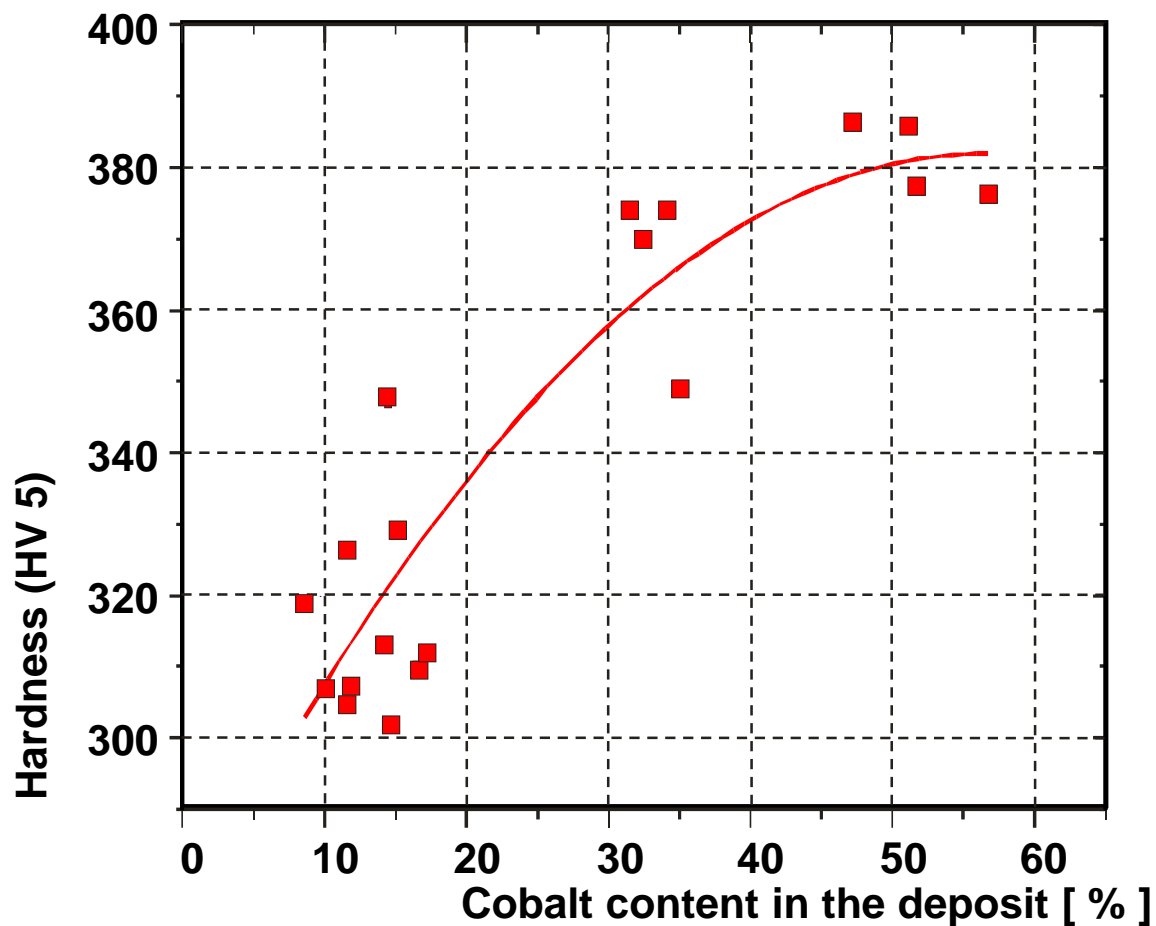


Cobalt content in the deposit versus cobalt concentration in the electrolyte

pH 4.0
T 55°C

Nickel	90 g/l
Sodium sulfamate	40 g/l
Sodium sulfate	15 g/l
Chloride	1.8 g/l

Electroplating of Ni-Co Microstructures



**Electrodeposition of Ni-Co micro-structures:
micro-hardness versus cobalt content in the deposit**

pH 4.0

T 55°C

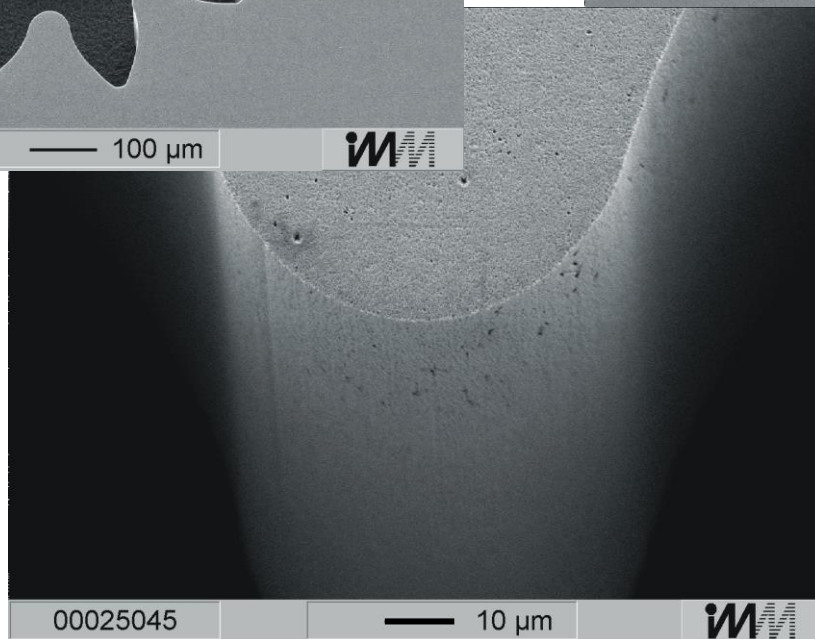
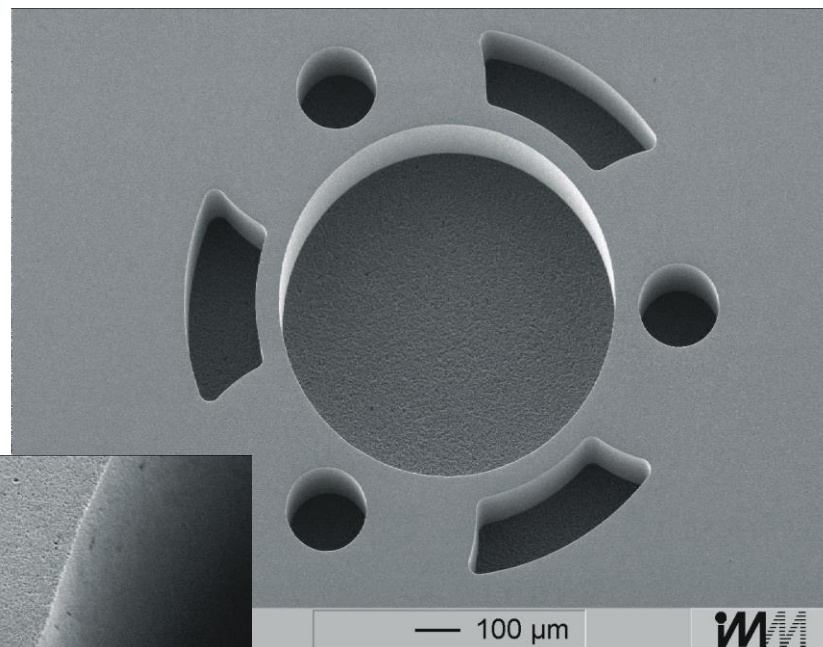
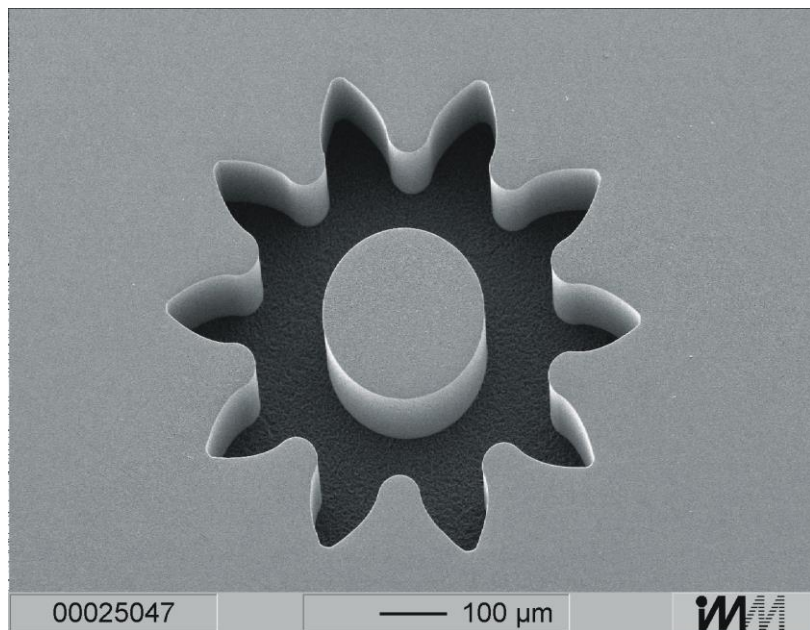
Nickel 90 g/l

Sodium sulfamate 40 g/l

Sodium sulfate 15 g/l

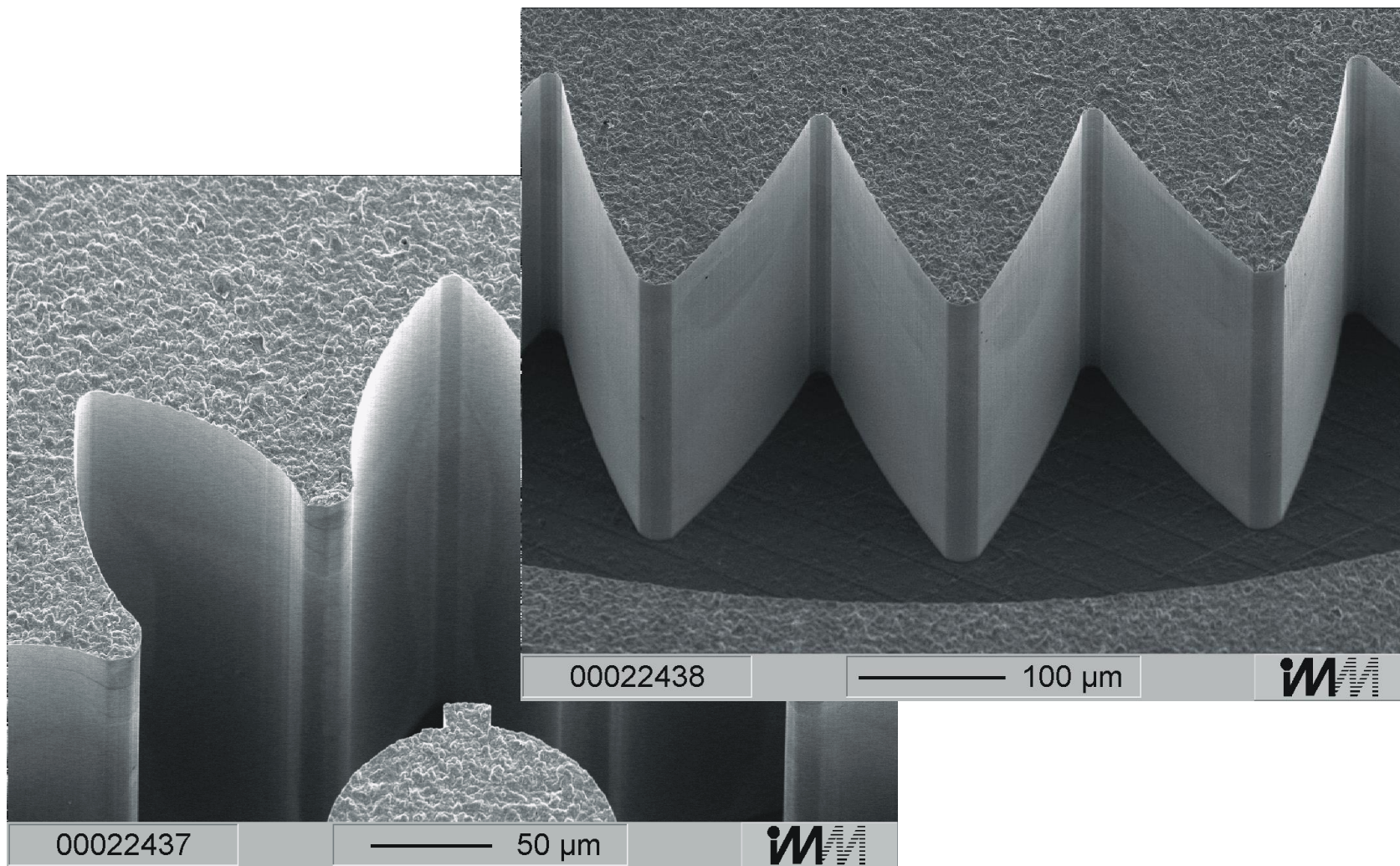
Chloride 1.8 g/l

Electroforming of Mould Inserts



Material: Ni-Co alloy

Electrodeposition of Ni-Co (73/27) Alloy



Electrodeposition

General

Gold

Copper

Nickel

Nickel-cobalt

Nickel- iron

Mold inserts

Special types of nickel alloys

Mold inserts from (ASE) silicon masters

Resist

Plating equipment

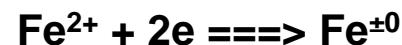
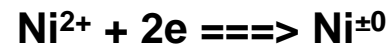
Nickel-Iron Alloy Electrodeposition: Working Conditions

Nickel	50 g/l
Iron	1-9 g/l
Boric acid	45 g/l
Sulfate	80 g/l
Complexing agent	15 g/l
Chloride	18 g/l
Saccharin	20 mg
Tenside	5 ml/l (2% Lösung)
Temperature	55 °C
pH - value	3.5
Current density	1 - 5 A/dm ²
Current efficiency	~ 98%
Volume	50 l

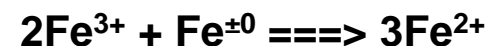
Anode reaction:



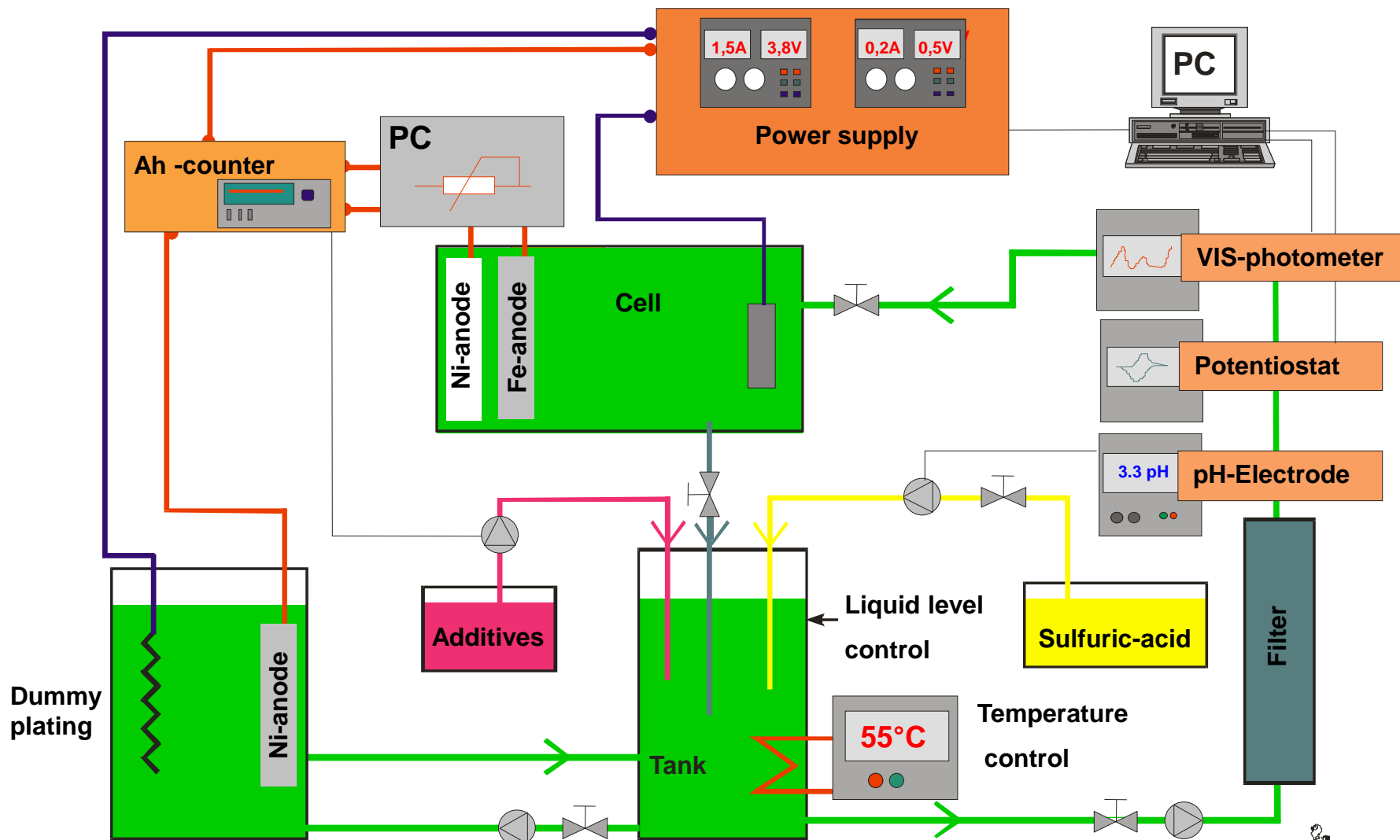
Cathode reaction:



Side reaction:

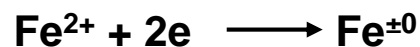
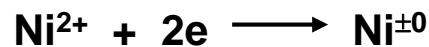


Nickel-Iron Alloy Electrodeposition: Flow Chart



Nickel-iron Sulfamate Electrolyte: Mass Balance

Cathodic reactions:



$j =$

$$j_{\text{Ni}} + j_{\text{Fe}} + j_{\text{H}}$$

Anodic reactions



Side reactions:



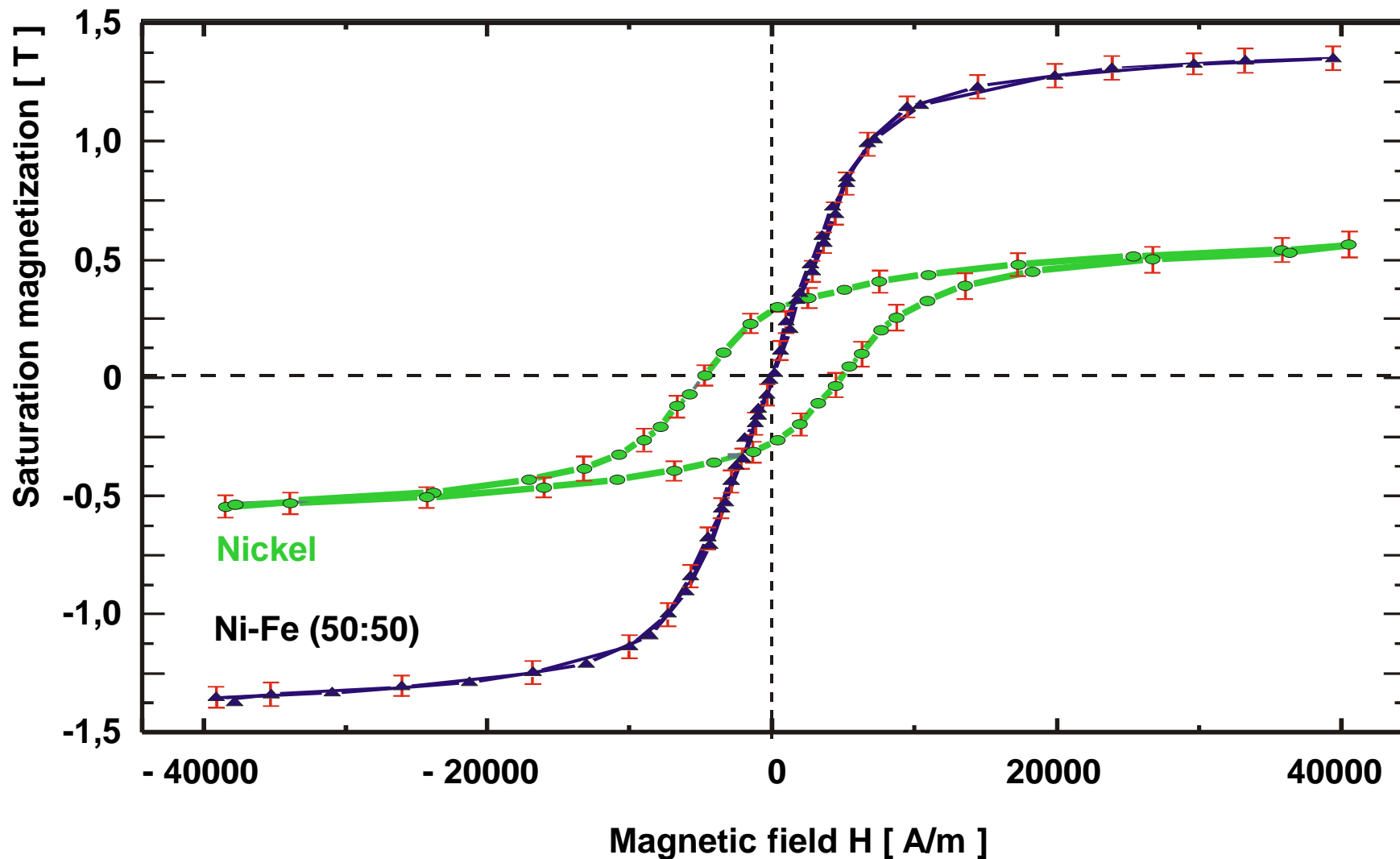
Regeneration cell



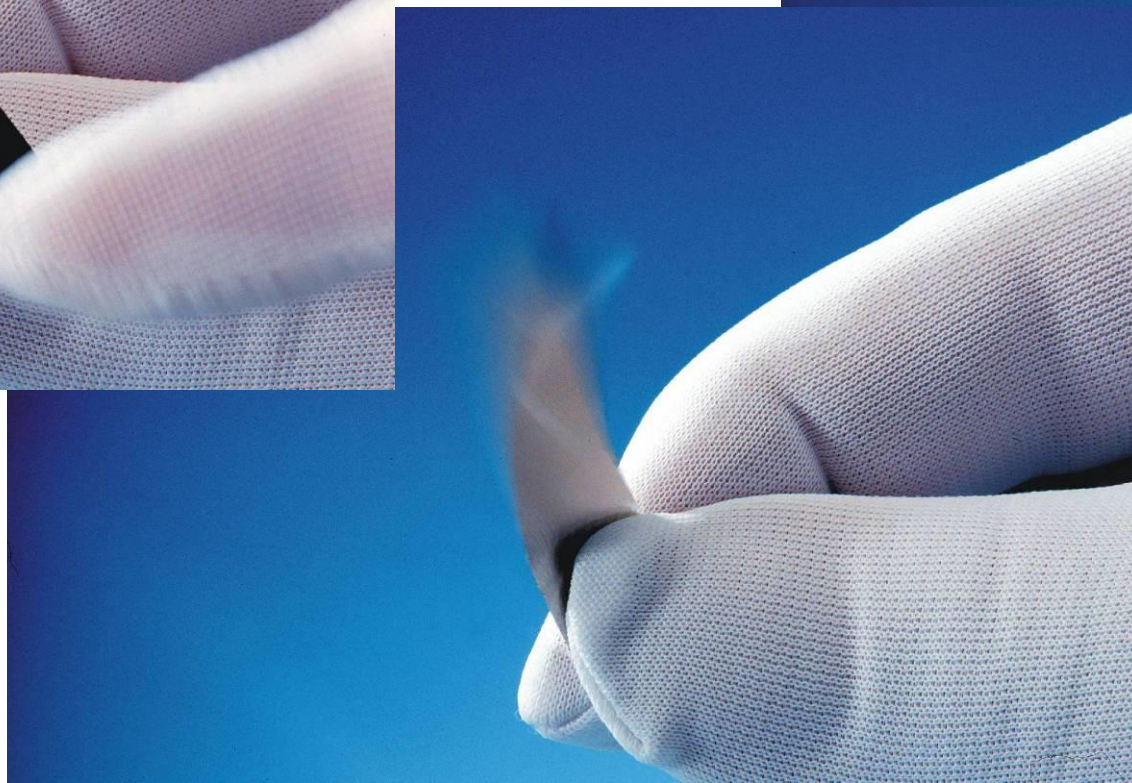
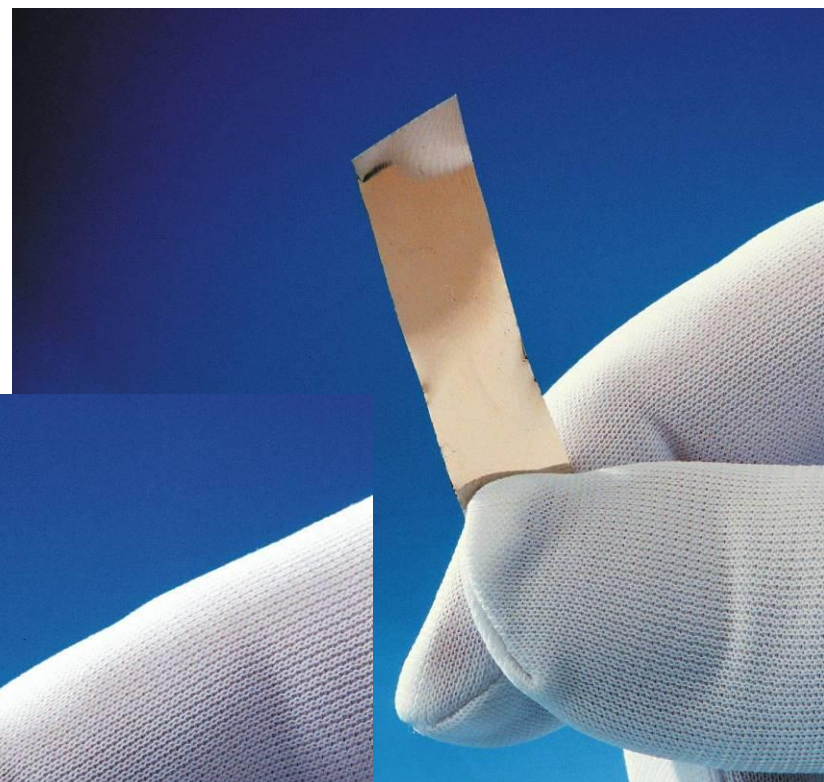
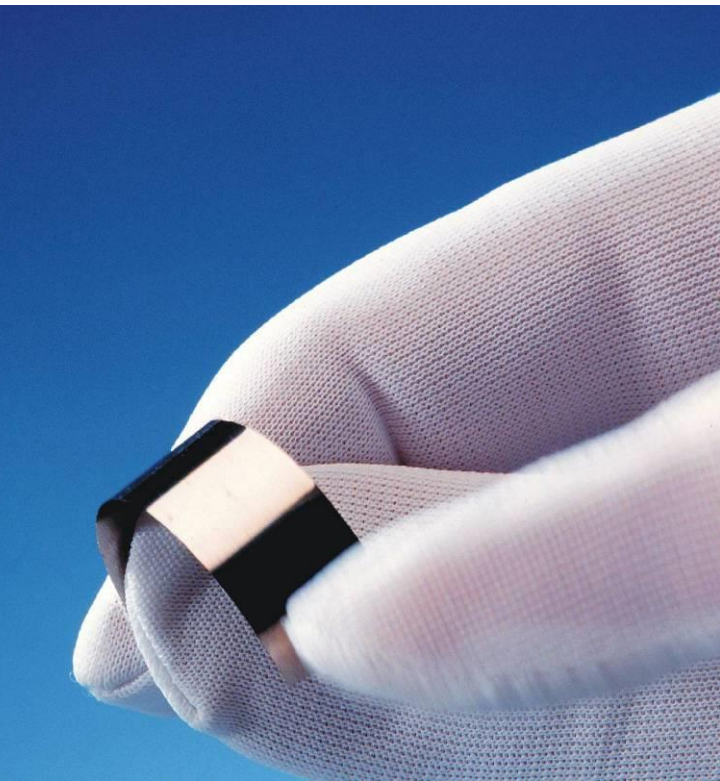
$$j_{\text{Ni}} + j_{\text{Fe}} + j_{\text{H}}$$



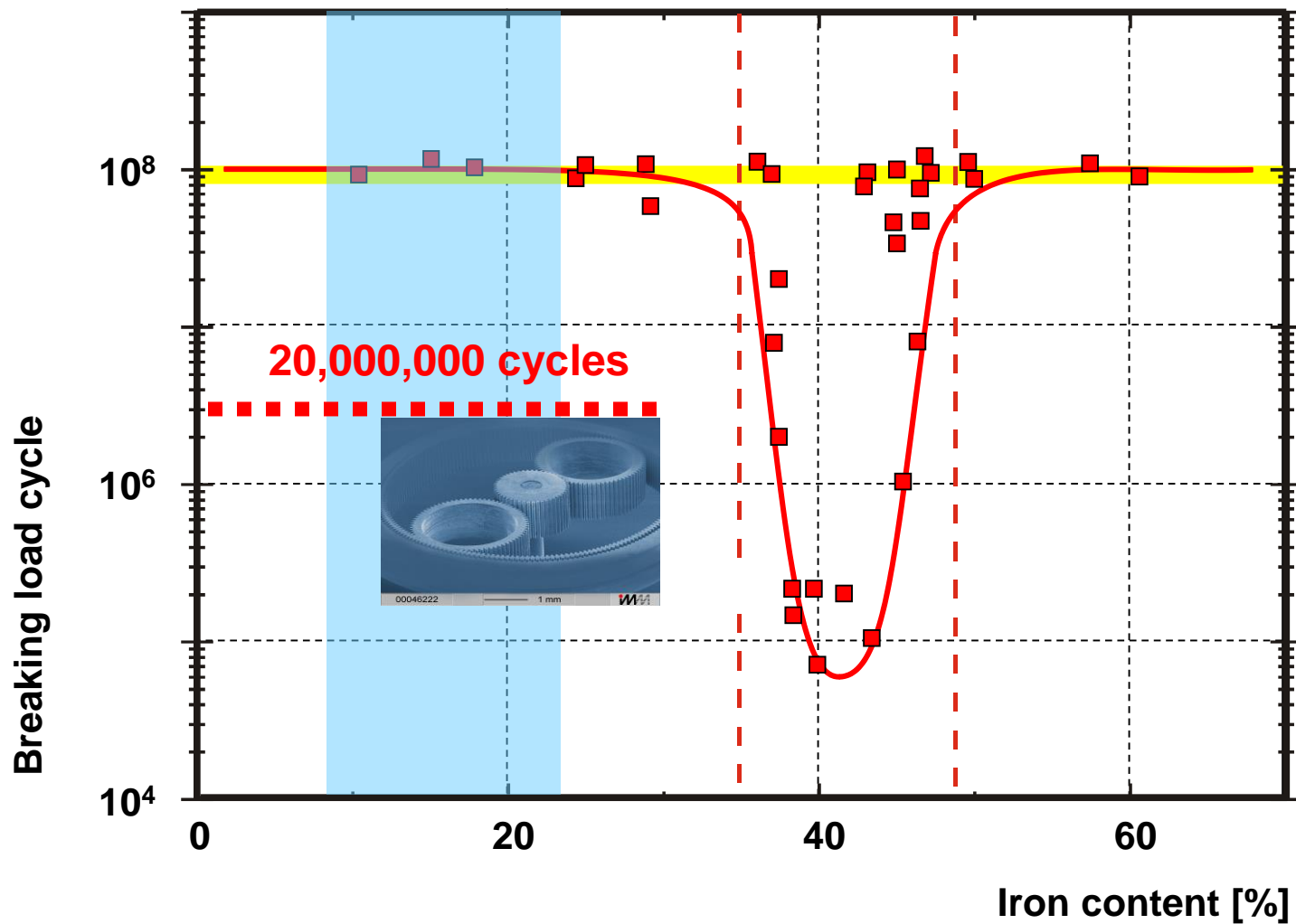
Comparison of Nickel and Nickel-Iron Microstructures: Hysteresis Curves



Material For Leaf-Springs; Electroplated Ni-Fe (90/10) Alloy

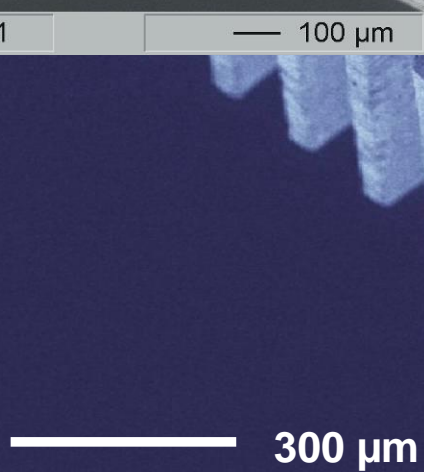
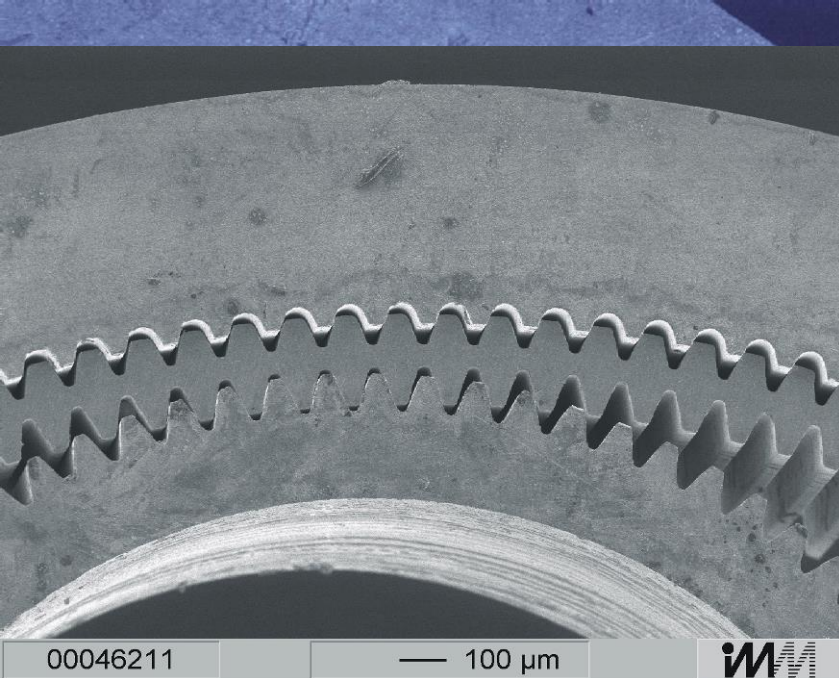


Endurance Strength of Ni-Fe Alloy Microstructures



Micro Harmonic Drive™: Flex Spline

- Gear ratio 500
- Torque 15 mNm
- Up to 50 000 rpm



Electrodeposition

Gold

Copper

Nickel

Nickel-cobalt

Nickel- iron

Mold inserts

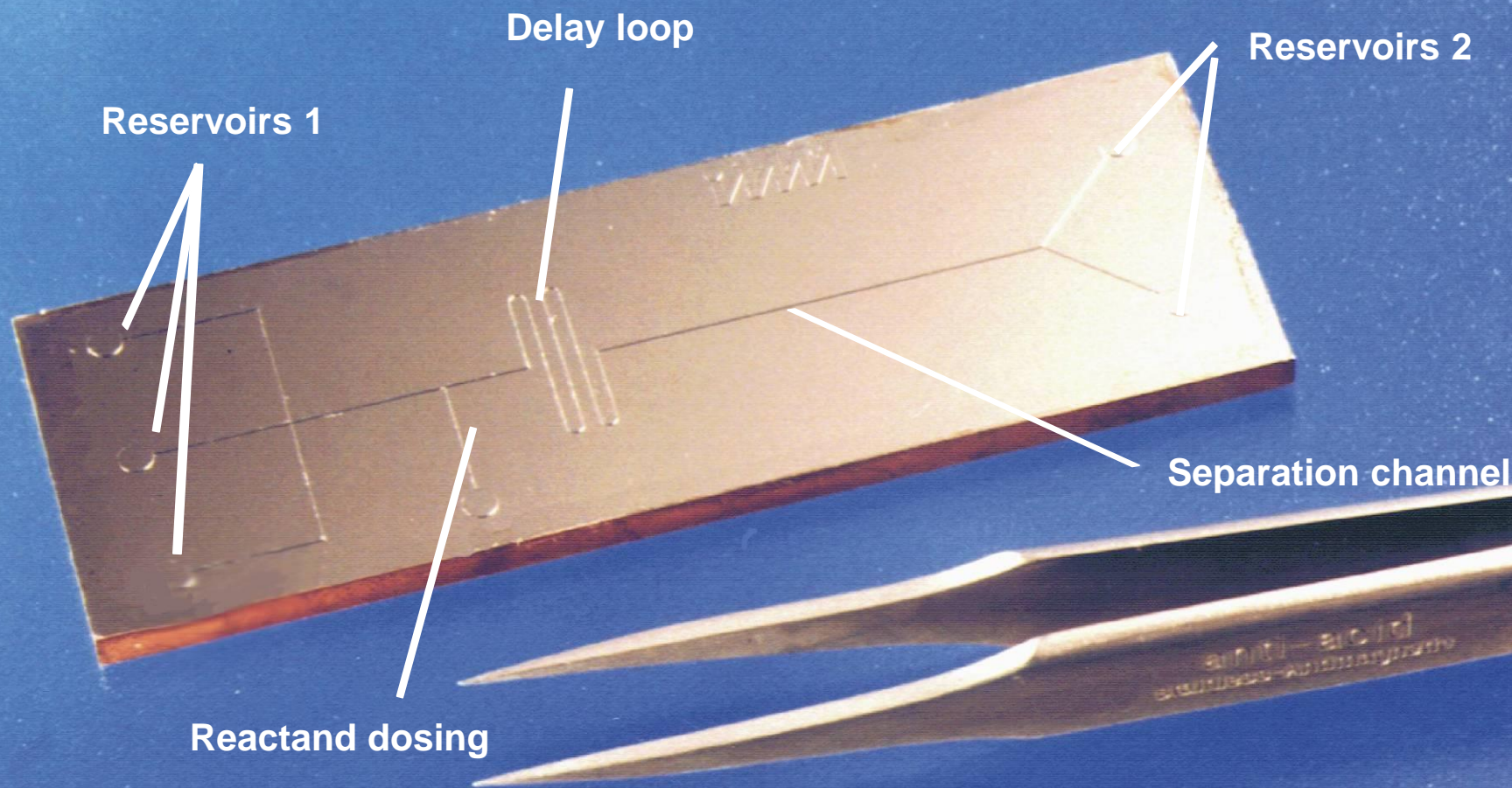
Special types of nickel alloys

Mold inserts from (ASE) silicon masters

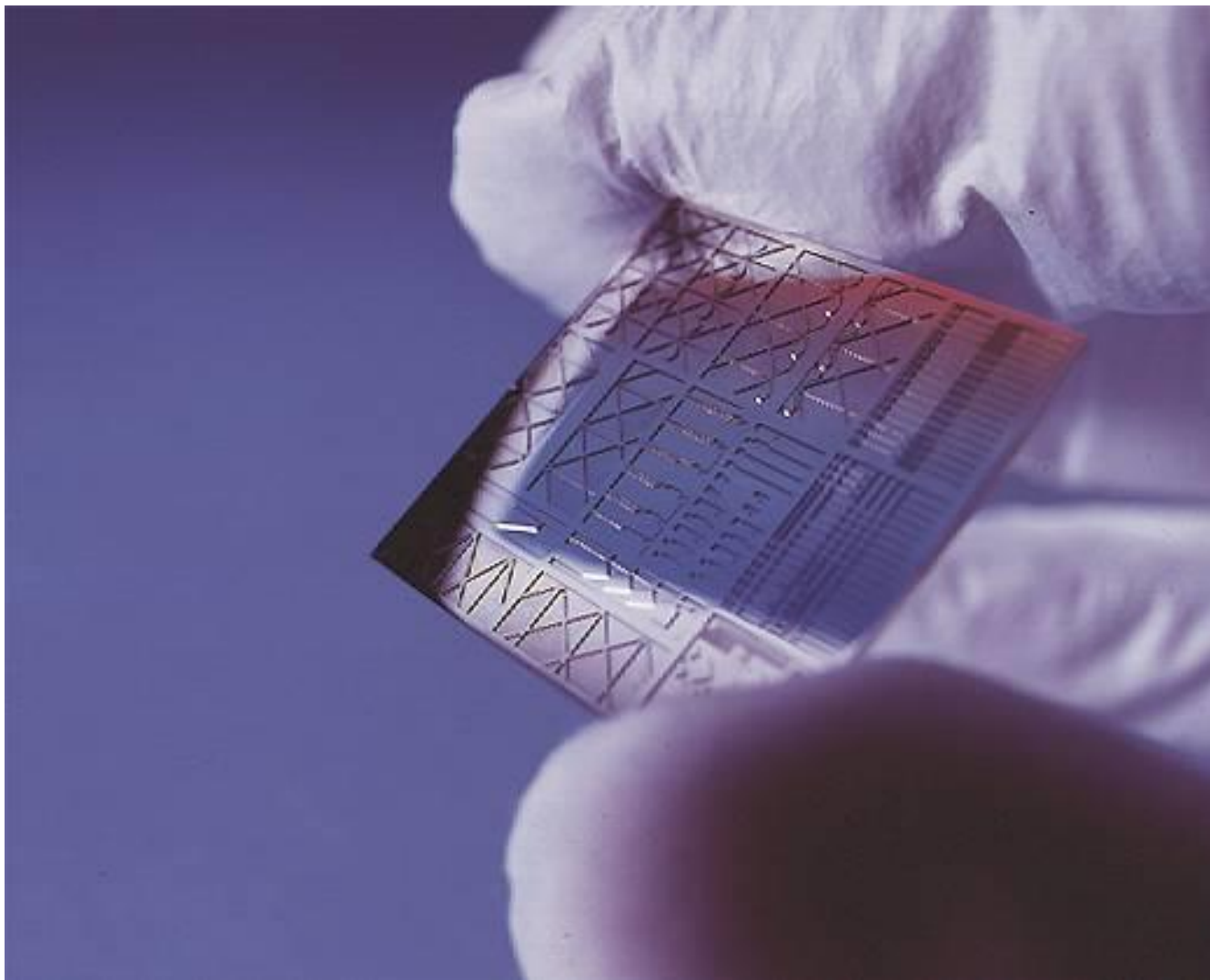
Resist

Plating equipment

Nickel Mold Insert for Lab-On-A-Chip Fabrication



Mold Insert for Micro-optical Structures



LIGA - technique

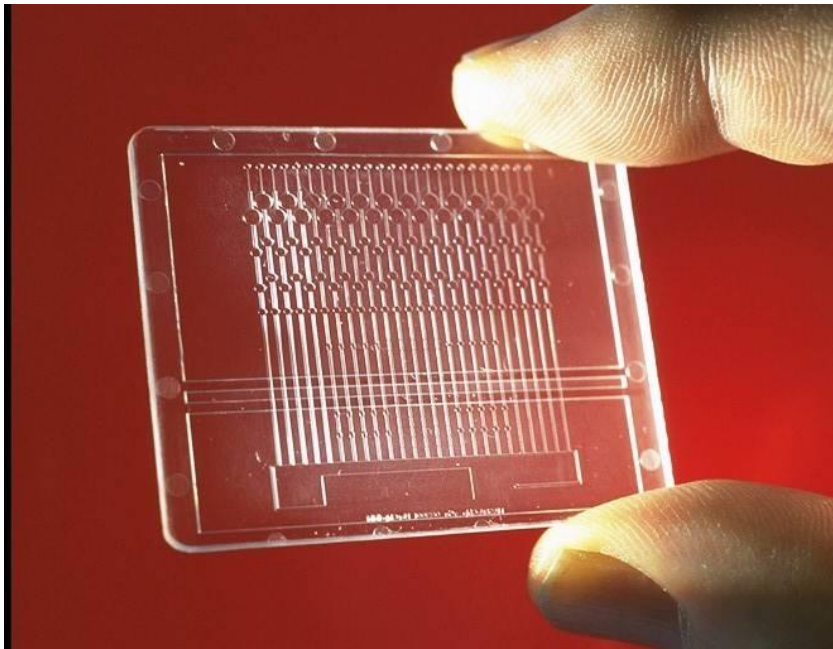
Material: Nickel

Dimensions:

25mm x 25mm

**Fiber Grooves
for 125 μm single-
module fibers,
microcuvettes and
lens systems for
beam shaping**

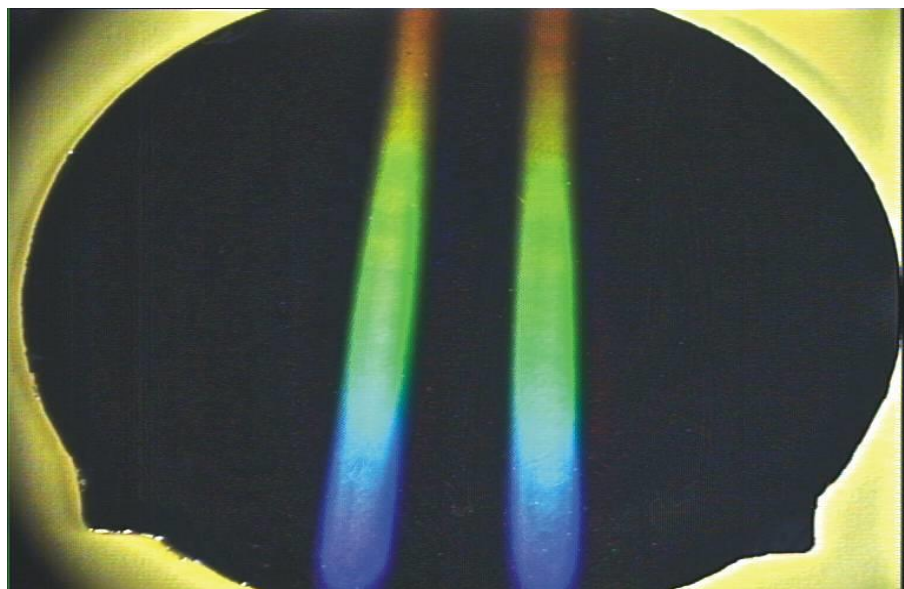
Polymer Chip Design and Fabrication



- 25 reaction chambers supplied by microfluidic channels
- Reaction volume 20 nl
- Liquid transport by combination of capillary forces and membrane actuators

- Fabrication of master by Advanced Silicon Etching (ASE)
- Electroforming of mold insert by using NiFe electrolyte
- Plastic chip manufacturing
- Surface functionalization of microchannels via wet chemical process

Ultraprecision Hot Embossing



Minimum feature size: <math><3\text{nm}</math>

Reproducibility: <math><1\text{nm}</math>

