MASS PRODUCTION OF THIN FOIL LASER TARGETS FOR USE ON NEXT GENERATION LASER FACILITIES

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OVERVIEW

- Background to high rep rate requirements
- Current Facility Requirements for HPL Targets
- The first RAL high rep rate experiment
- Developments in target mass production
- The Nano-positioning wheel
- First fabrication runs
- Flexibility vs. target numbers
- Finalised design and next stages.

SCOPE

2008 - high power laser shots < targets available

2010 - high power shots >> targets available

What to do with extra shots?

How do we delivery targets to these shots?

Applications based experiments – security, medical, IFE?

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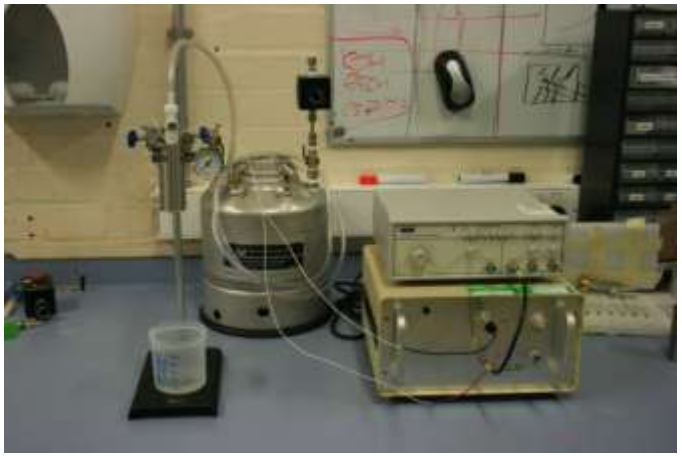
The increasing number of high rep-rate, high power laser systems in the world can run at rates of up to 10Hz.

For 10 Hz it may be necessary to use (established) real-time target delivery techniques

- Tapes (including multilayer)
- Structured tapes
- Droplets
- Gas jets



Tape speed ~ 5mm/sec

Tape substrates - Copper, PE, Al, PET (Mylar)

R&D Programme to produce multi-layer tapes

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In continuous operation at 16Hz one IFE reactor will require:

~ 1 500 000 targets per day

Future systems such as ELI will be running in the kHz regime.

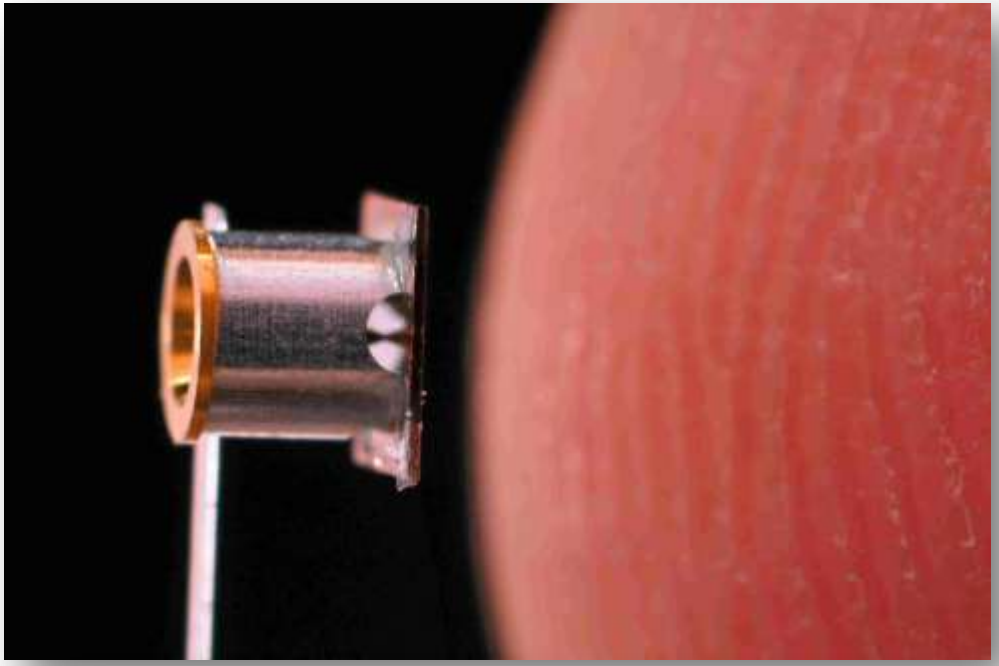
Targets for these experiments cannot be produced using current techniques and so new technologies need to be implemented.

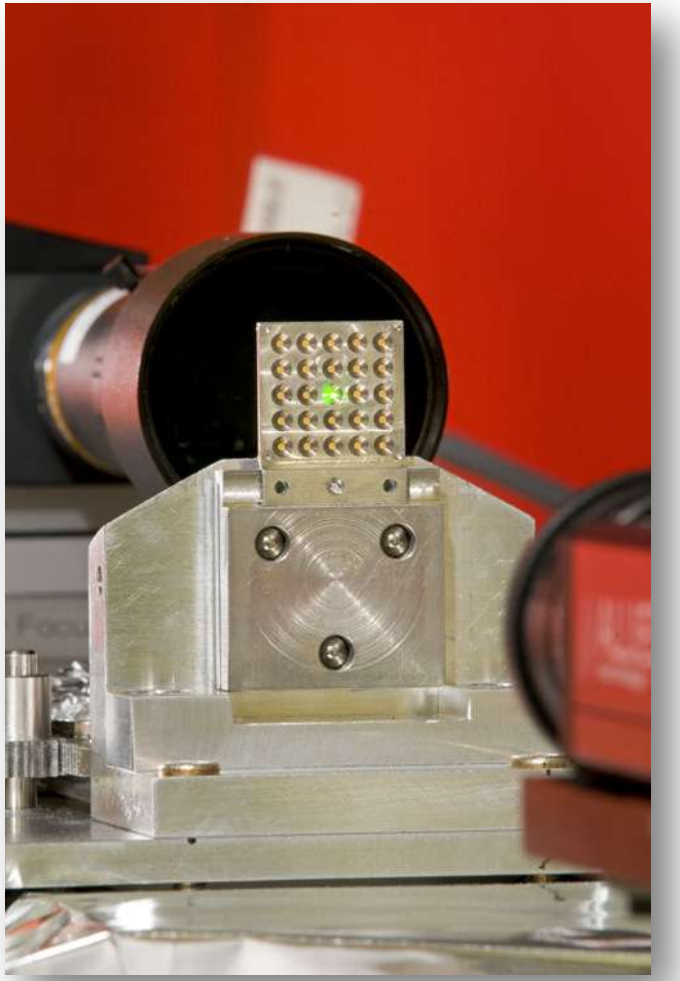
One possible solution to this is to use MEMS technology integrated with injection/insertion systems.

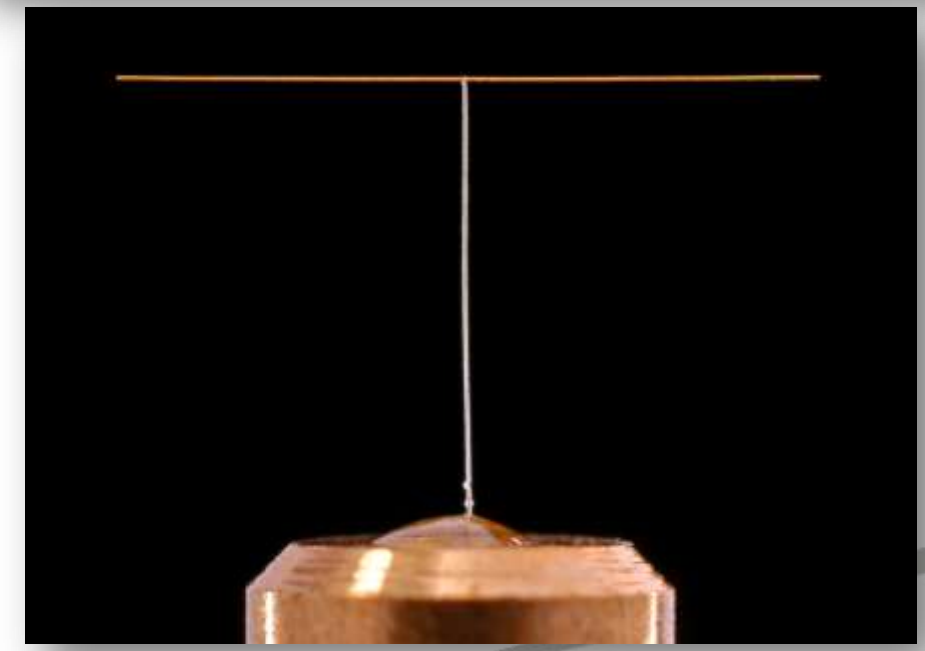
GENERAL TARGET TYPES

It is difficult to define categories or groups for high power laser targets as they are inherently different for each type of experiment. However the can be loosely grouped into the following.

- Simple Thick Foil Targets (>1um)
- Thin Foil Targets (20nm-1um)
- Ultra Thin Foils (<20nm)
- Multi-layer or complex films
- Single components 3D targets
- Multi-component 3D targets
- Multi-target arrays







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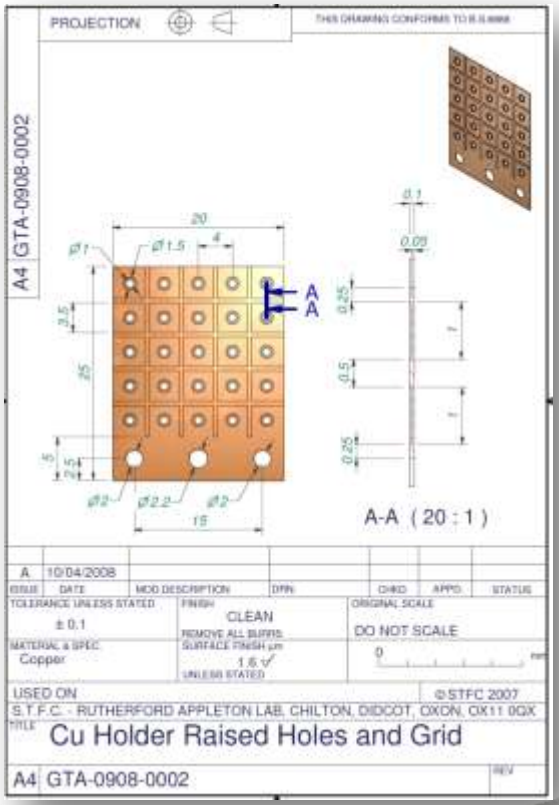
APPLICATIONS BASED TARGETS

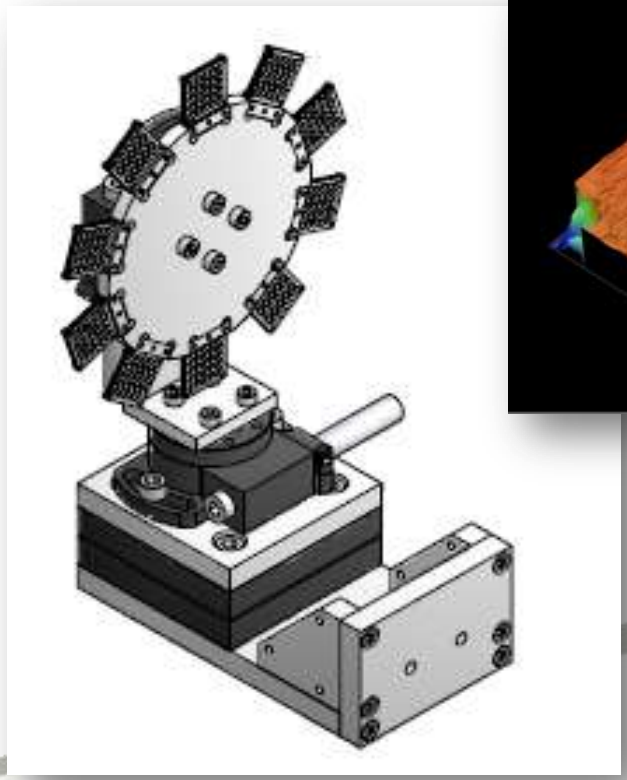
The first HRR solid target experiment at RAL shot over 3000 targets, most of these were thick but the interesting science is carried out using the ultra thin foils, using thin foils for statistical data and using more complex targets.

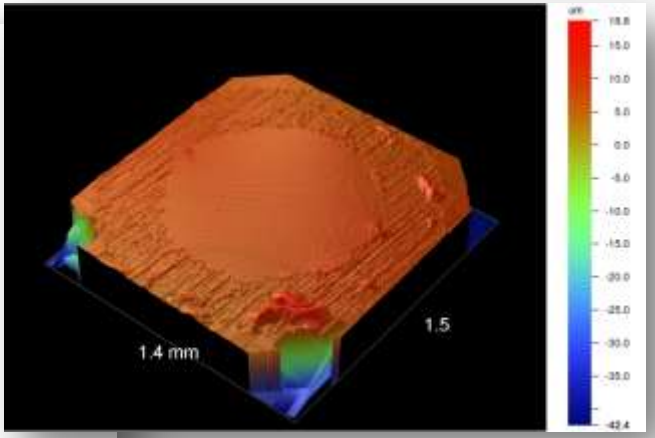
These complex targets are possible to make but are more difficult to fabricate for higher rep-rates. Examples of these are:-

- Ultra thin foils
- Ultra thin foams
- Micro-spoke targets
- Wire arrays
- Limited mass (100um x 100um squares).

The first HRR experiments were delivered using a ‘puck’ and wheel arrangement. This is labour intensive to deliver as each foil has to be floated onto a mount, characterised logged and then attached to the wheel. While the quality of the foils was good, there are a limited number of shots that are available to the user group and as you move to thinner foils (<20nm) the yield of the targets on a ‘puck’ is low.







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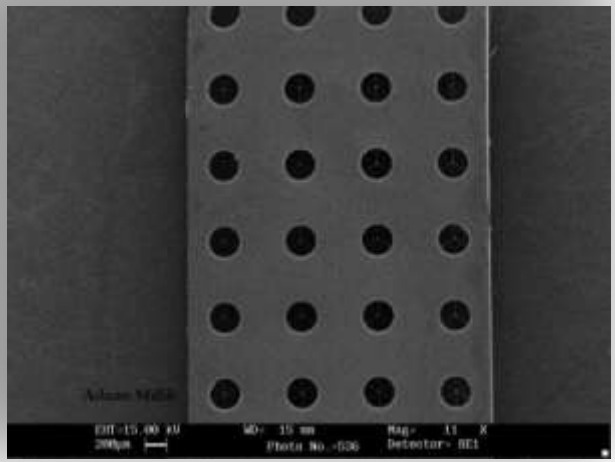
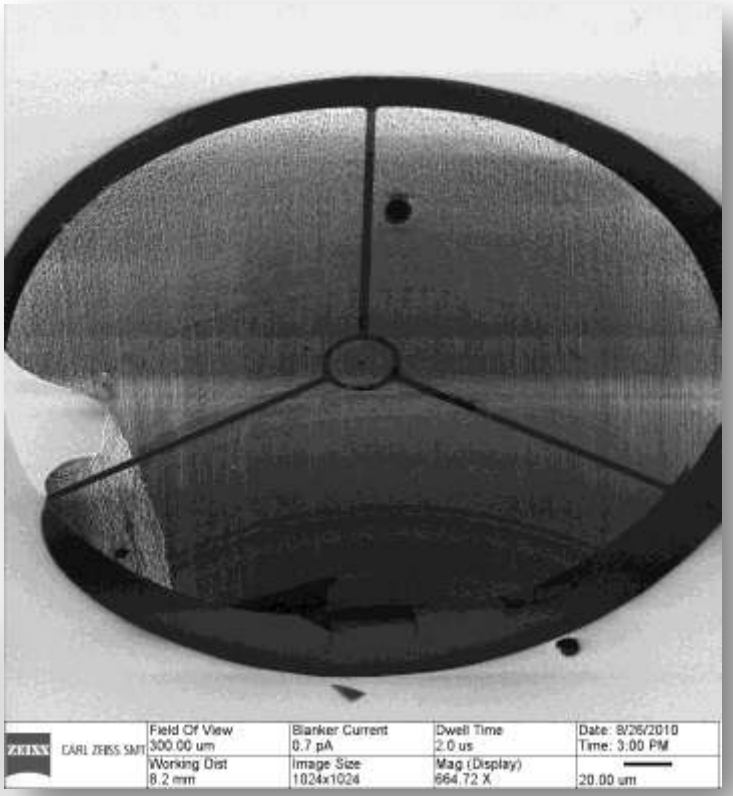
MASS-PRODUCTION DEVELOPMENTS

There have been a number of developments in mass-production of targets, including using CNC milling machines to batch produce parts.

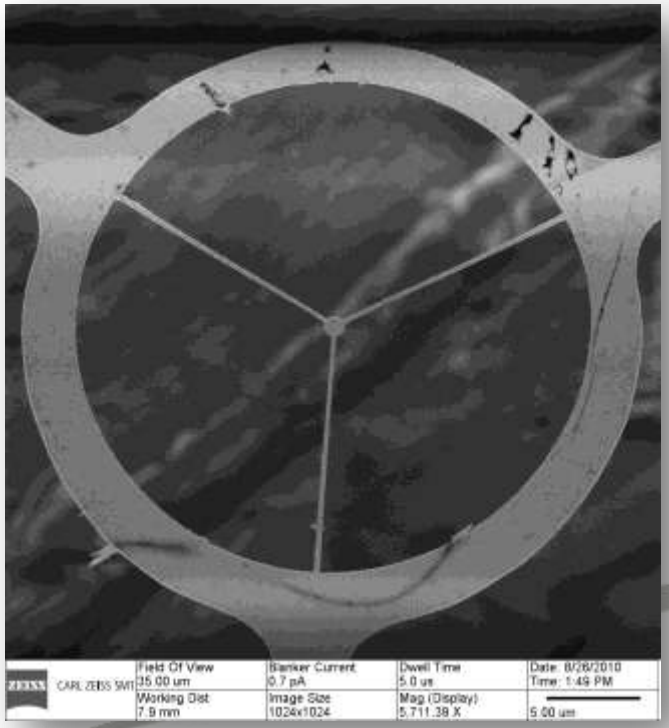
However, the only realistic way of producing targets for the HRR laser systems of the future is to use MEMS (Micro-electro-mechanical systems). This has advantages of large volume manufacture with high based fabrication yields and allows targets that cannot be conventionally machined to be fabricated

RAL has produced 2D and 2½ D components in the past using these techniques and this could be developed to provide a targetry stream for all HRR laser systems that is cost effective.





Membrane targets - 32um diameter, 40nm thick SiN membranes supported on 1µm wide, 40nm thick arms



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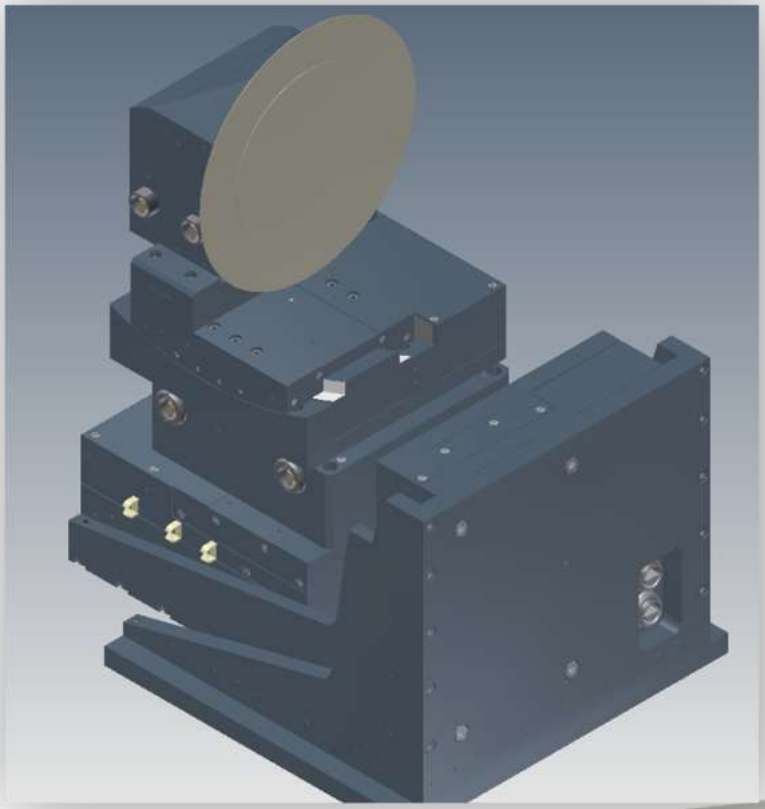
NANO-POSITIONING WHEEL

A design for a high specification target wheel with 6 axis translations a small footprint and high accuracy drives was produced. The wheel can hold 4” or 6” wafers.



- 3 Linear Translations
  - Travel = 60mm
- 3 Rotational Translations
  - 2 X Travel = 360˚
  - 1 X Travel = 30˚
- Small Footprint
  - 230mm x 260mm
- Encoded Resolution
  - 20nm

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NANO-POSITIONING WHEEL

The targetry solution for the wheel are designed to achieve the following aims.

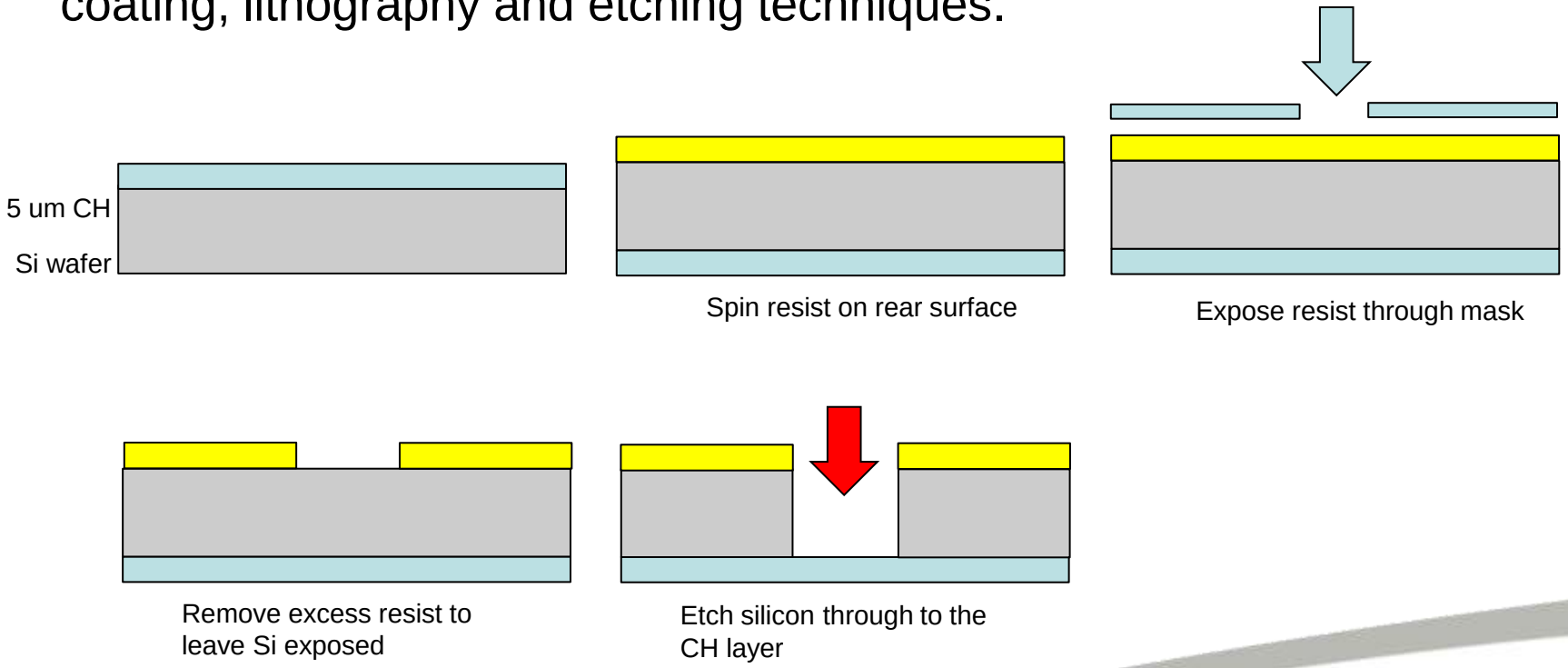
- Standard design Si wafers can be used
- Standard geometries of target layout can be used
- Recipes for variation of hole separation etc.
- Recipes for variation of foil material and thickness.

Essentially this is a ‘off the shelf’ target that can be used for a variety of both application and science based experiments.

It is also essential that 1000’s of targets could be fielded onto a target wheel to take advantage of the high repetition rate of the latest generation lasers and also to make the wheel experimentally feasible.

TARGETRY DEVELOPMENT

To deliver a robust targetry solution to the wheel 4” or 6” wafers need to be processed to delivery 1000’s of targets on a single wafer. Using MEMS techniques a target can be produced on a wafer using coating, lithography and etching techniques.



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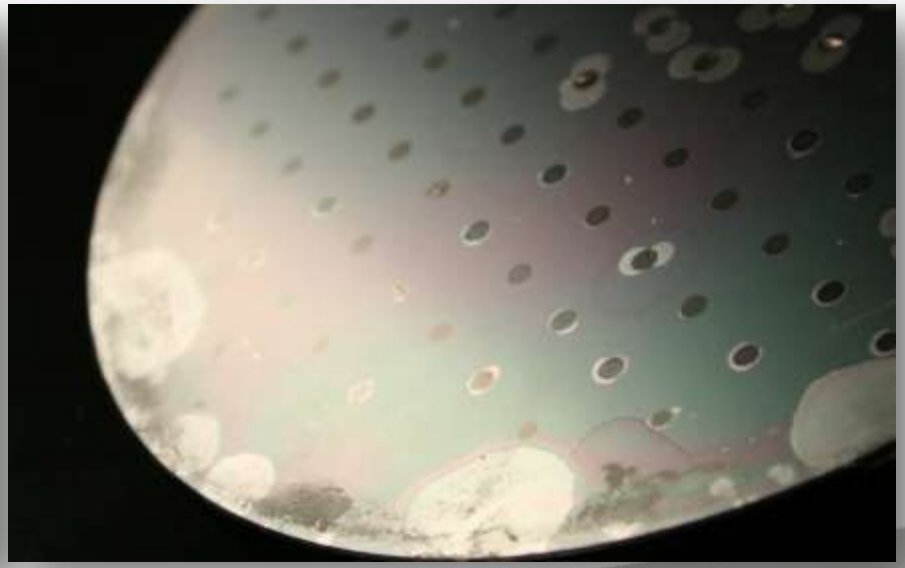
TARGETRY DEVELOPMENT

This technique was used to pattern a 100mm wafer with a number of holes. A 5um Parylene layer was used for the target foil.

A 100% yield was achieved with the foil produced exhibiting excellent flatness. (Dependent on the initial substrate roughness)



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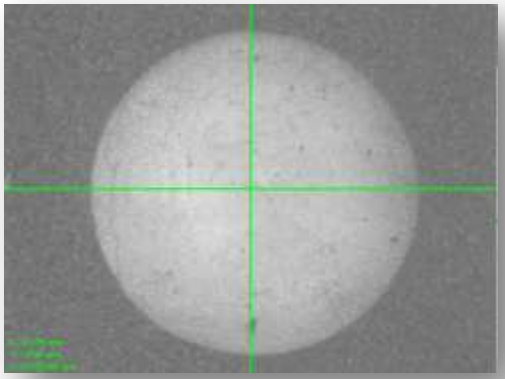
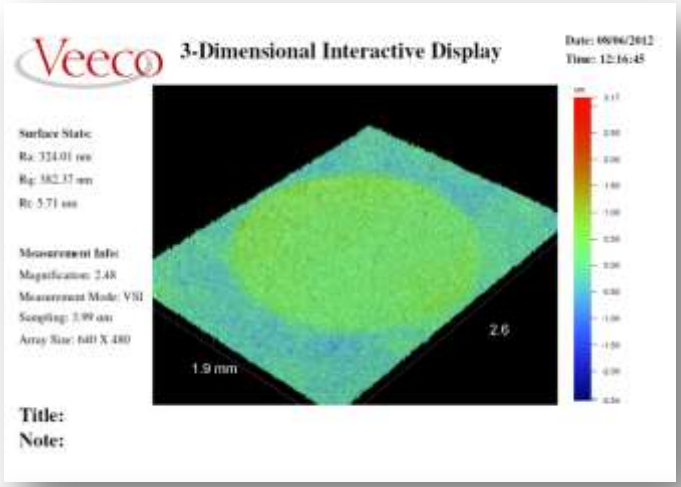
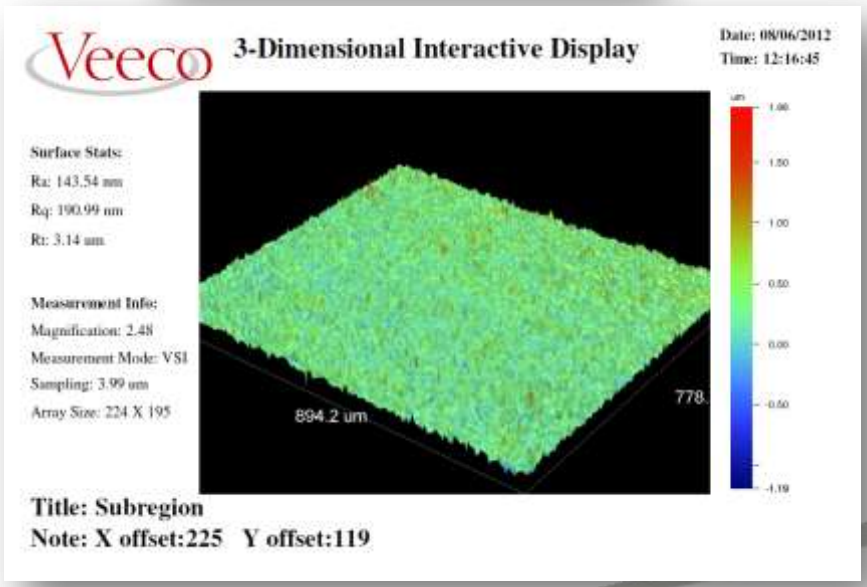
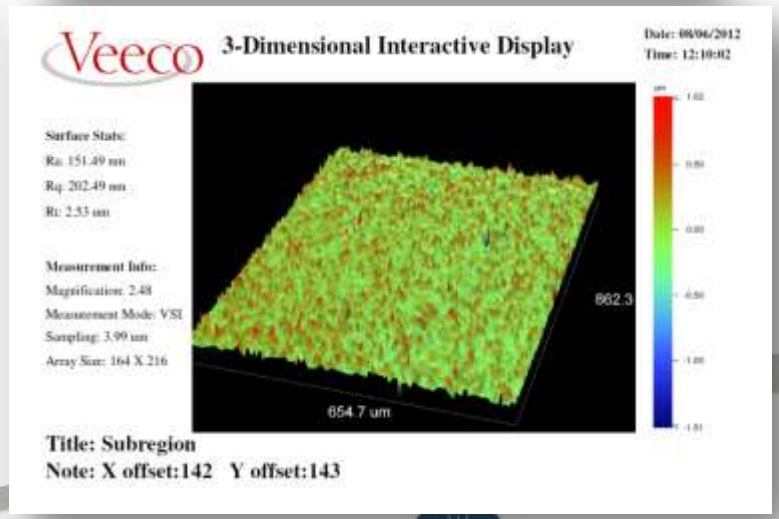
TARGETRY DEVELOPMENT

The surface roughness of the film is similar to the surface roughness of the unpolished Si wafer.









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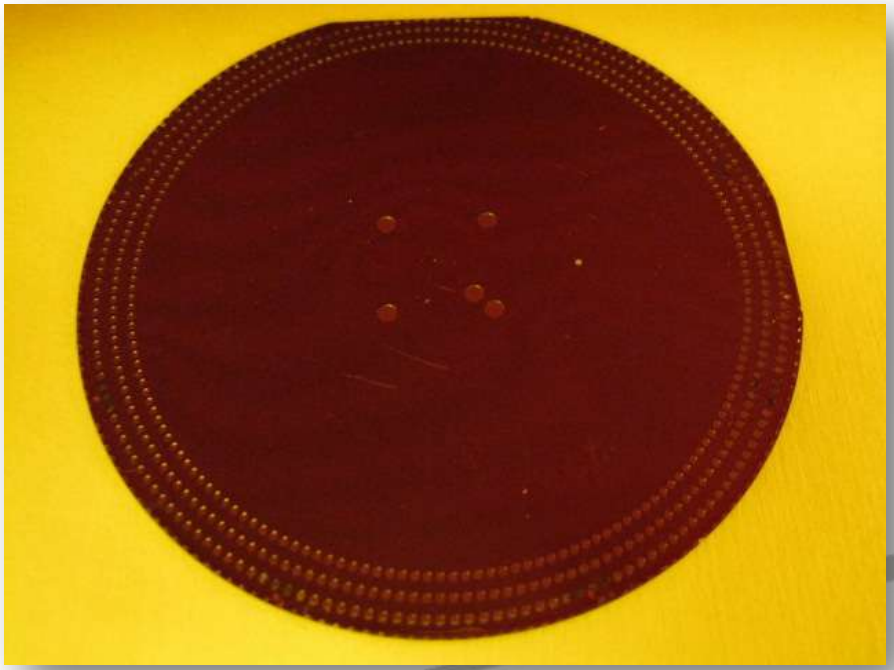
TARGETRY DEVELOPMENT

A mask was produced to allow the wafer to be fitted to the wheel and also to pattern a large number of targets around the circumference. The material used for the target was 50nm SiN and a similar etching process was used to the previous targets.

Although successful the yield of the targets was low – due to etching variations across the diameter of the wafer.



- 3 concentric rings of ~500 thin film targets
- 50nm thick silicon nitride
- 500μm diameter
- 300μm thick wafer

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FLEXIBILITY VS. TARGET NUMBERS

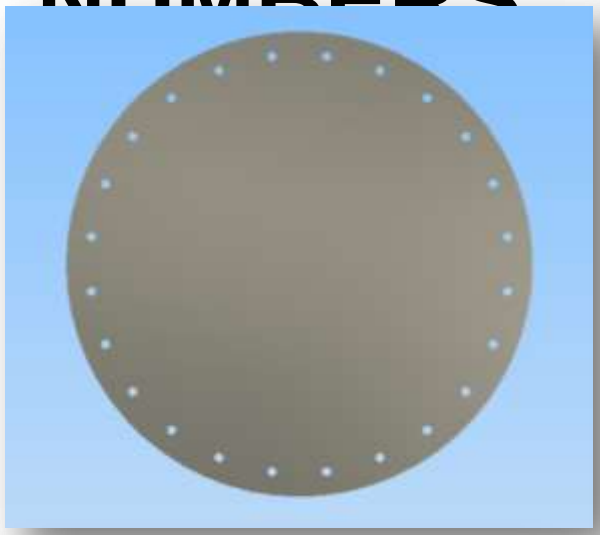
There is an inherent problem with a single silicon target piece as a component for the wheel. This is that only one target type can really be supplied per wheel.

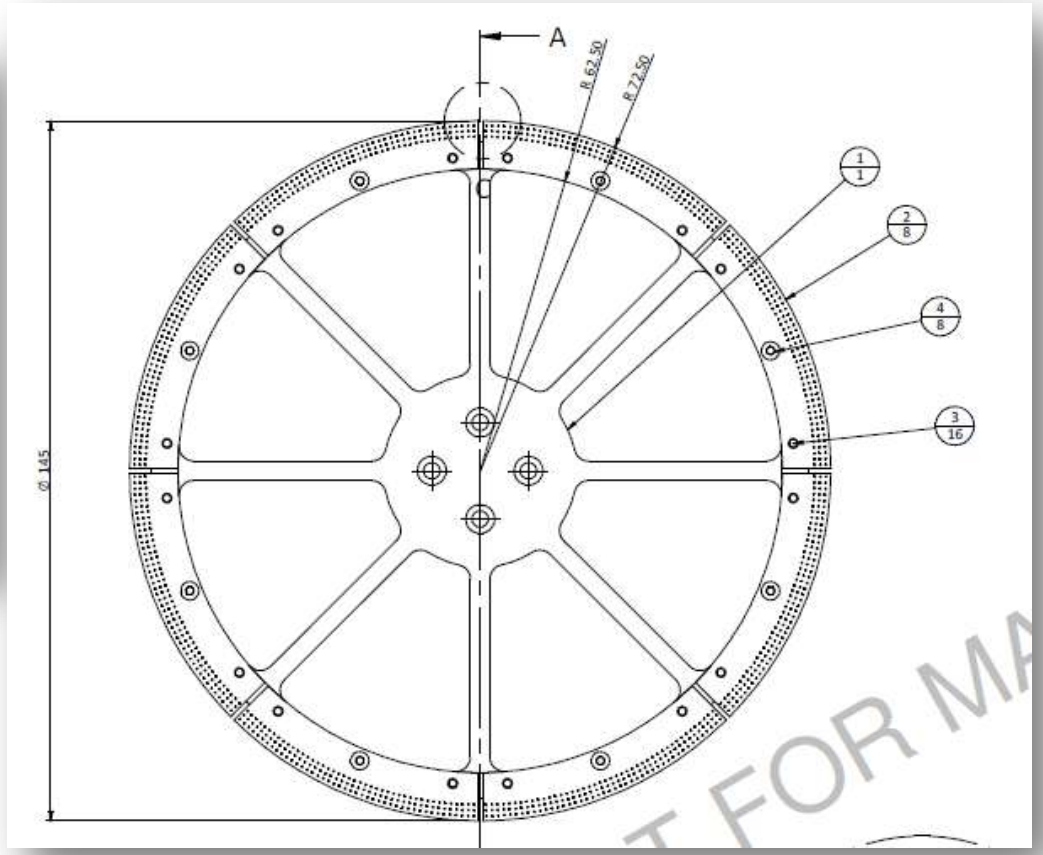
Experience has shown that the ideal experiment has a large number of targets but also variation in parameters such as material, foil thickness.

It was therefore decided to split the wheel into sections and to be able to deliver ‘slices’ of wafers onto a holding ring.

This approach still delivers a mass produced target that can be ‘off the shelf’ but gives experimental flexibility and will allow maximum use of laser time which can be up to 4x oversubscribed.

FLEXIBILITY VS. TARGET NUMBERS





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COMPLETED WHEEL SECTIONS

The completed wheel sections

Protection layers are removed using a number of dry and wet etching techniques

Yield from a 100mm wafer is almost 100% for 5um thick CH foils.

Foil roughness has been reduced to levels comparable to float off techniques.

Sections can be attached to the ring as required for each experiment.

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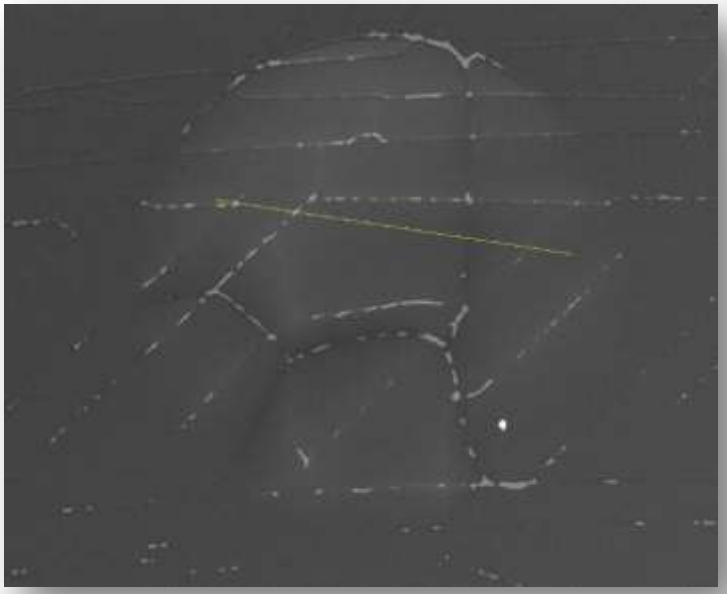
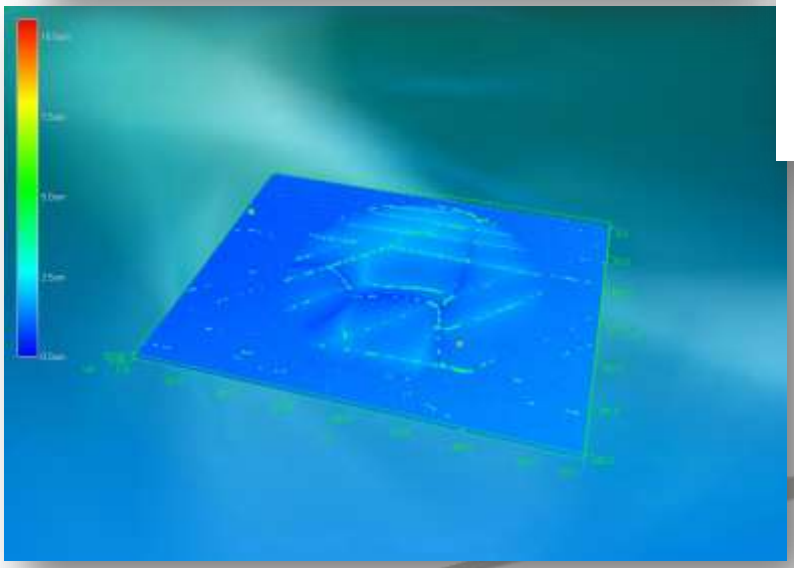
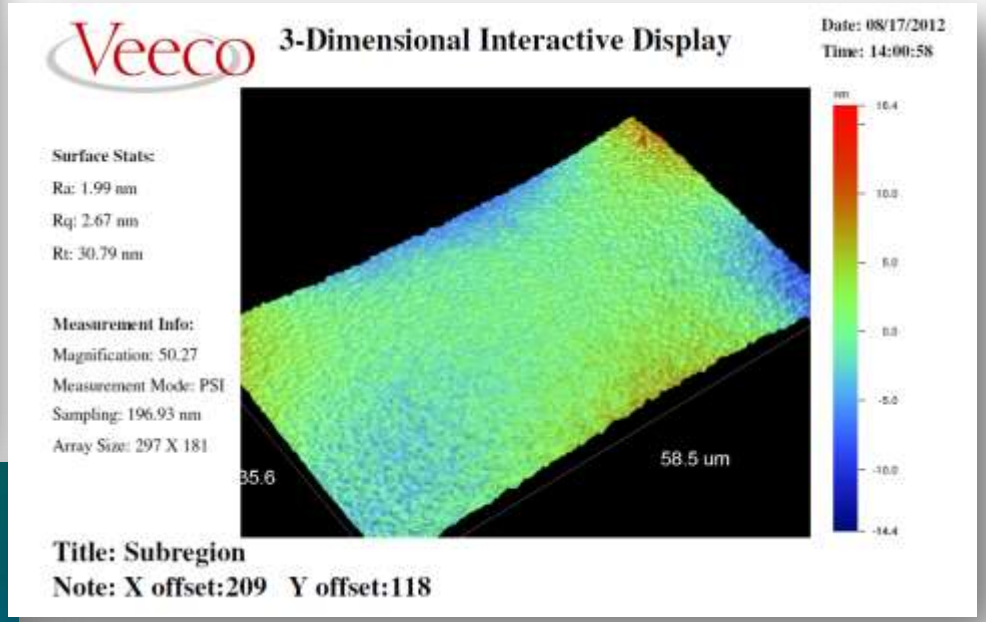
COMPLETED WHEEL SECTIONS







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COMPLETED POSITIONING WHEEL

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NEXT STAGES

Further work is required to establish production techniques to:

- Produce thinner films
- Measure wheel repeatability
- Investigate shock damage
- Integrate more complex targets

However initial trials have shown that simple thin foil targets can be produced in high numbers and the nano-positioning wheel has the ability to be a flexible method of delivering these targets to an experiment.

THANK YOU

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Fabrication of Mass Produced Microdot Arrays for use as Micro-Targets on High-Repetition Rate Experiments.

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Outline

1. Background/Motivation
2. Target Design
3. Fabrication
   - Basic processes
   - Special Precautions
   - Putting it all together
4. Conclusions
5. Questions

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Background

- Small target size
  - In cases where the is focused beam on a large target, the range of intensities may cover many orders of magnitude (e.g. FEL x-ray source)
  - Micro-scale target intercepts the beam at specific intensities
- Large arrays
  - Where high repetition rates are used
  - MEMS-based fabrication allows 10s – 1000s to be easily fabricated in precisely defined arrays

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Target Design

- Micro-dot size
  - Typically a few microns in diameter
  - Thickness up to a few microns – experiment/material dependent
  - Each dot is supported on a thin, low-Z (CH) membrane over a cavity in the support substrate
- Array size
  - Typically 40 x 40 (1600 dots)
  - Potentially could be much larger (e.g. 100 x 100 or more)

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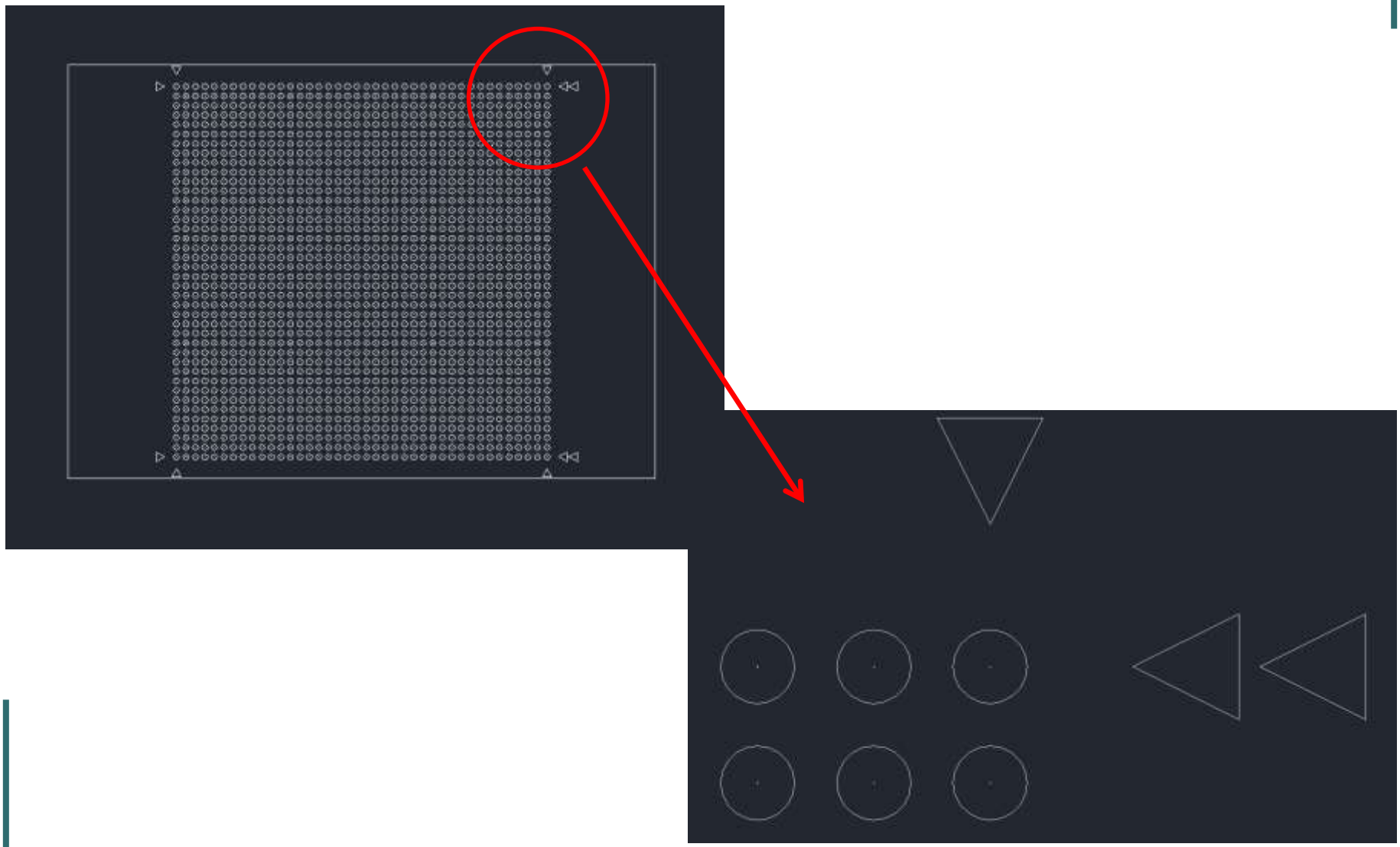

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Fabrication

- MEMS-based fabrication processes
  - Substrate
    - (usually) silicon wafer
  - Patterning
    - Microlithography (optical or e-beam)
  - Deposition
    - sputter, thermal evaporation, CVD, electro-deposition, spin/dip-coating
  - Etching
    - dry (plasma) etching, wet etching

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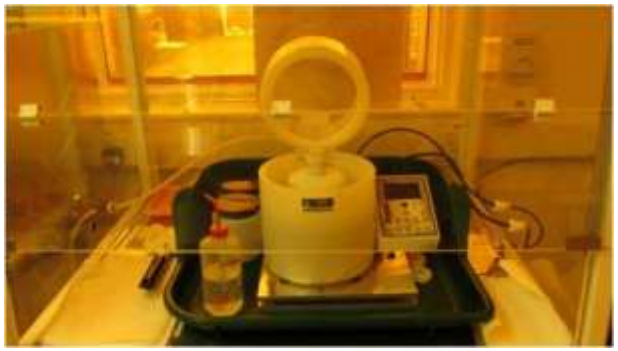

Spin-coater

Mask aligner

E-beam

DRIE

RIE

Stepper

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Special Precautions

- Semiconductor manufacturers use a relatively limited range of materials
  - Silicon, aluminium, gold, chromium, copper, ...
- Laser Target Fabrication requires a much wider range of materials
  - Nd, Sm, Te, CsI, Sn, Au, Cu, Bi, Ag, Gd, Fe, etc, etc.
- To ensure compatibility during processing, it may be necessary to add extra masking layers, e.g.,
  - Standard Gold etch solution also etches iron
  - Adhesion of metallic coatings on polymers or Si

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Putting it all together

Example Fe microdots as laser targets

- **Fe dot targets on a suspended CH film**
  - Fe dots - Diameter = 5 um diameter
    - Thickness = 2 um
  - CH film - Thickness = 1 um
  - Substrate - Silicon
    - Cavity diameter = 350um
    - Array size = 40 x 40 (1600 targets)

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Single-side polished silicon wafer. (~400um thick)

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Deposit Parylene Layer (1 micron)

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Deposit adhesion layer (approx 5nm)

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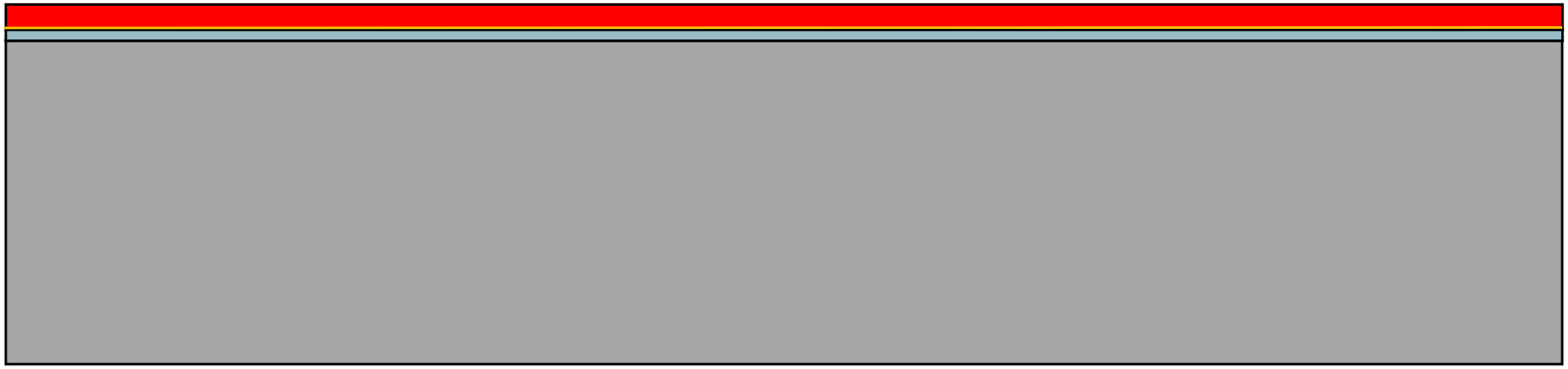
Deposit bottom LOR resist layer (thickness to suit dot thickness)



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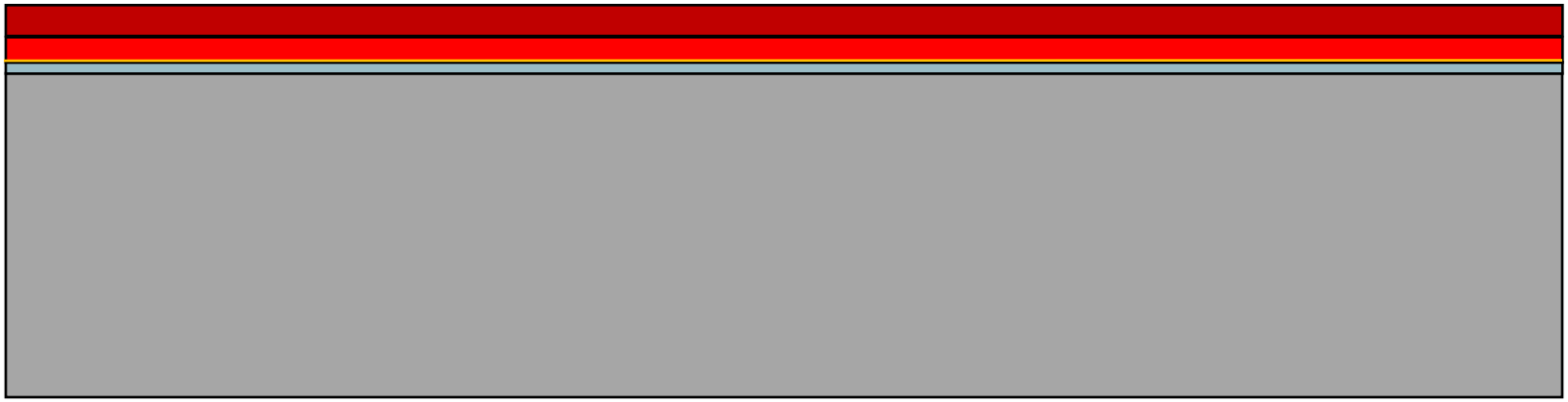
Deposit top imaging resist layer (~1um)



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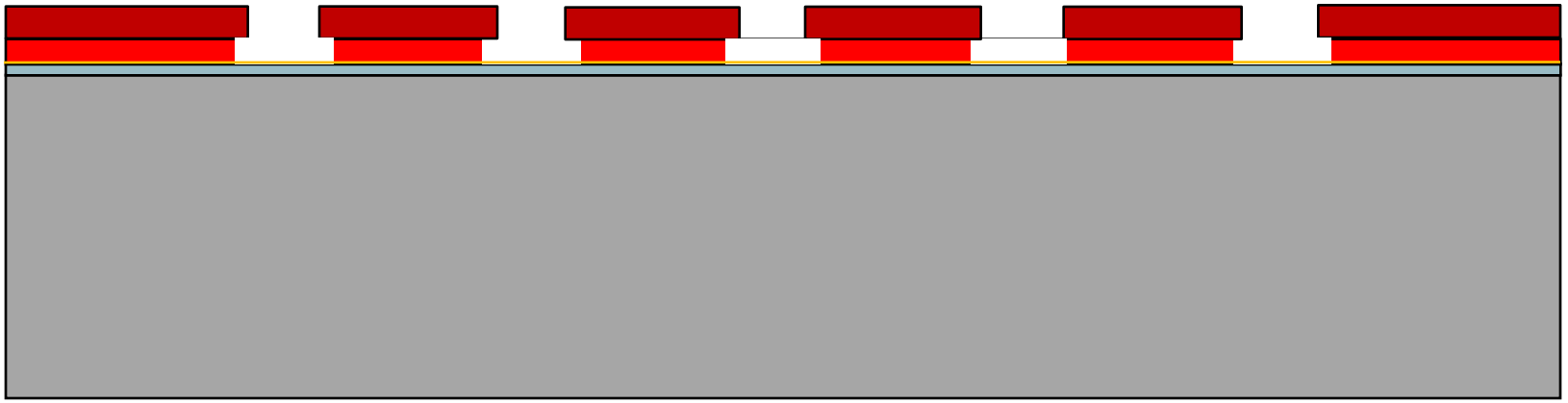
Expose & develop bilayer holes



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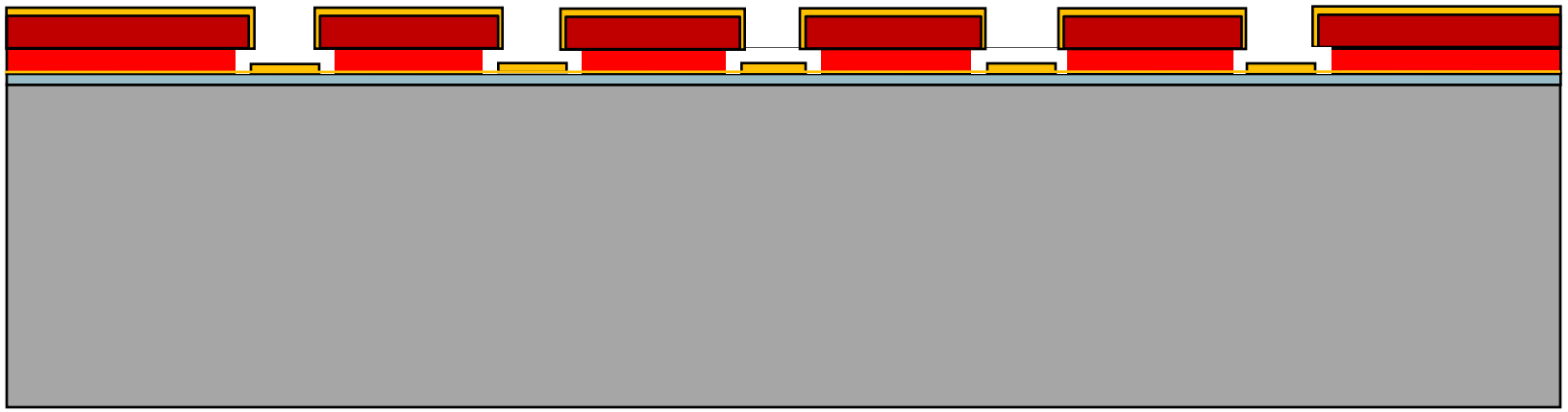
Thermal deposition of Fe layer (1-2um)



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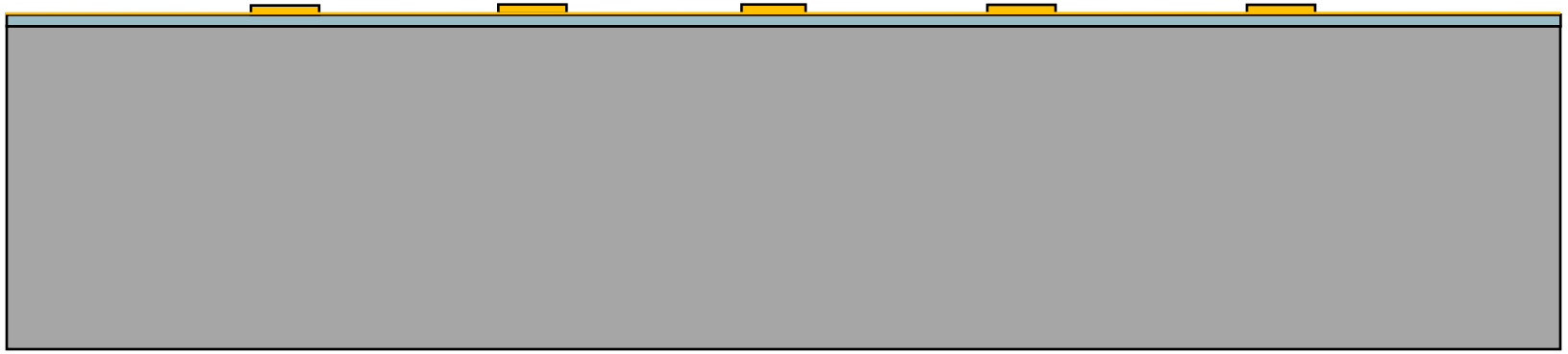
Lift-off resist to leave metal dots



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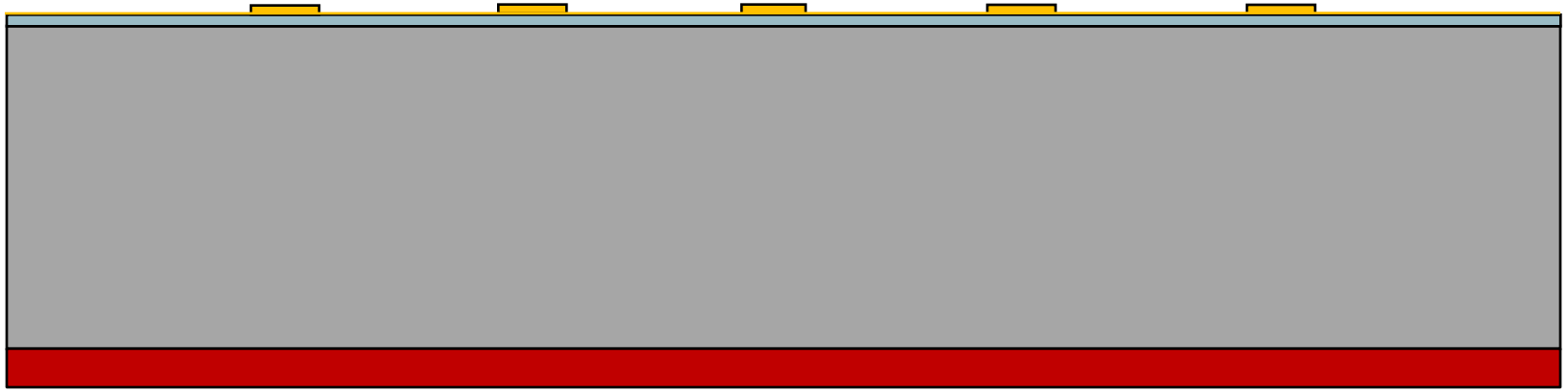
Spin-coat DRIE resist on lower surface



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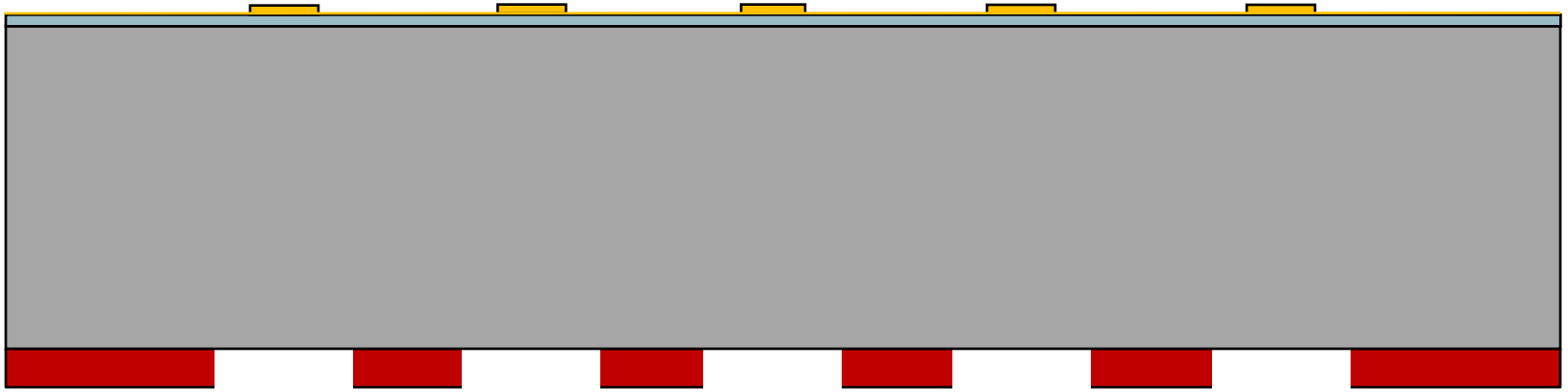
Expose and develop resist



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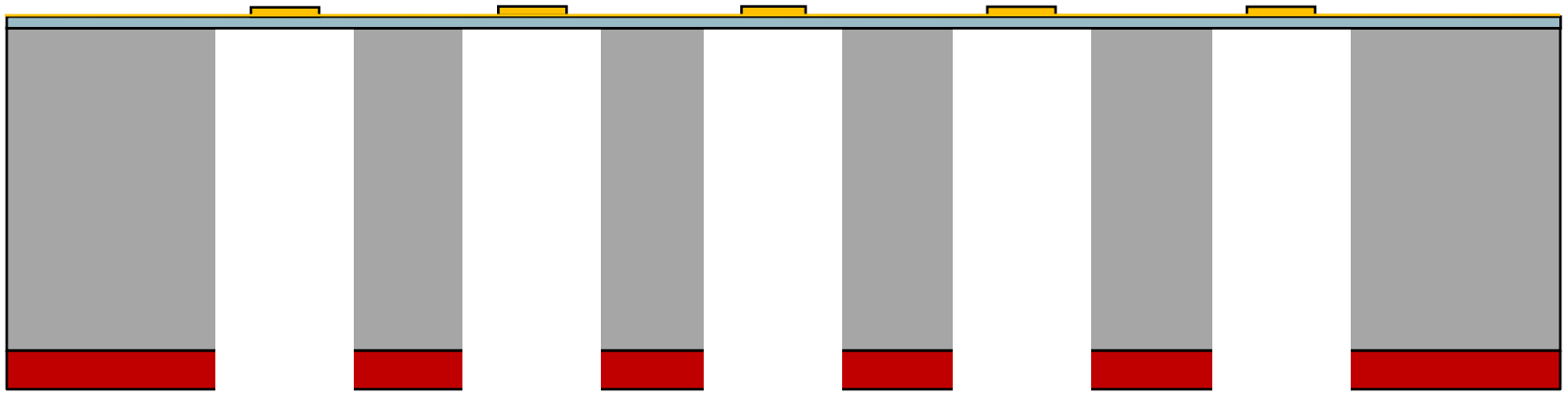
DRIE – Etch through to parylene (also dices substrate)



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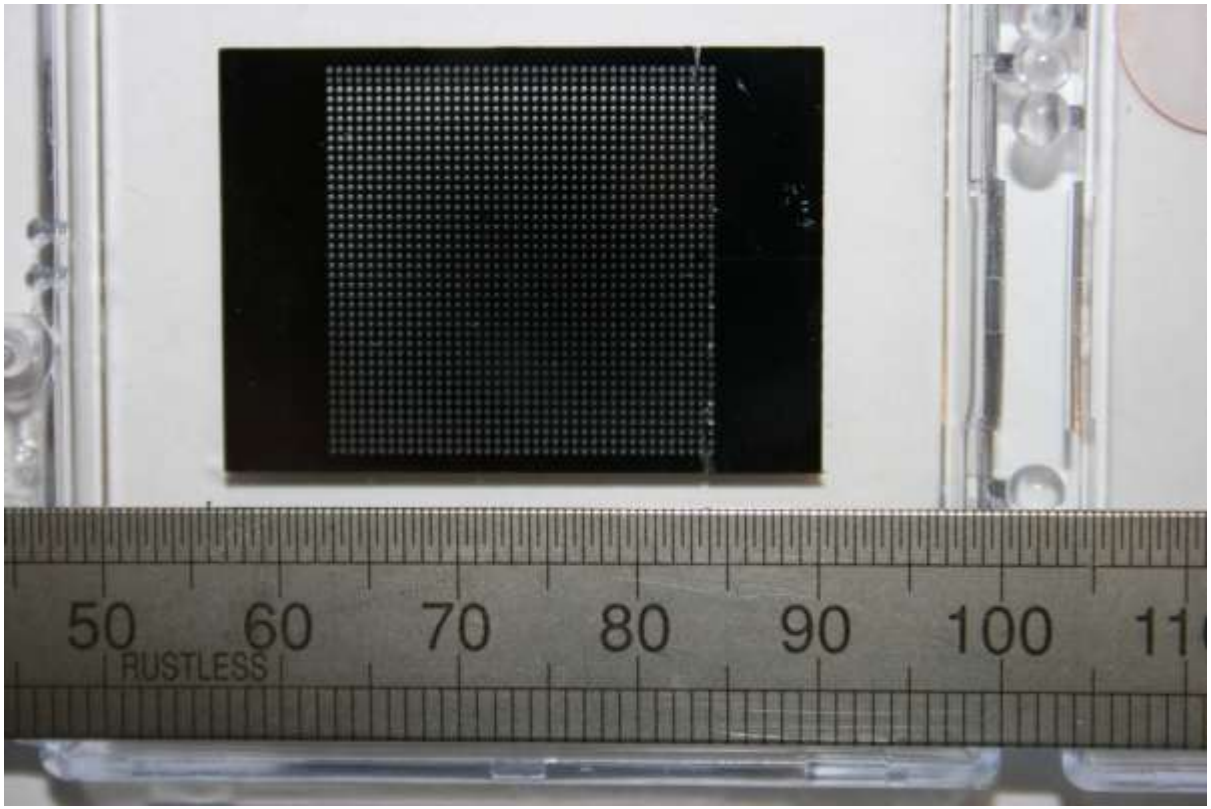
Visual inspection



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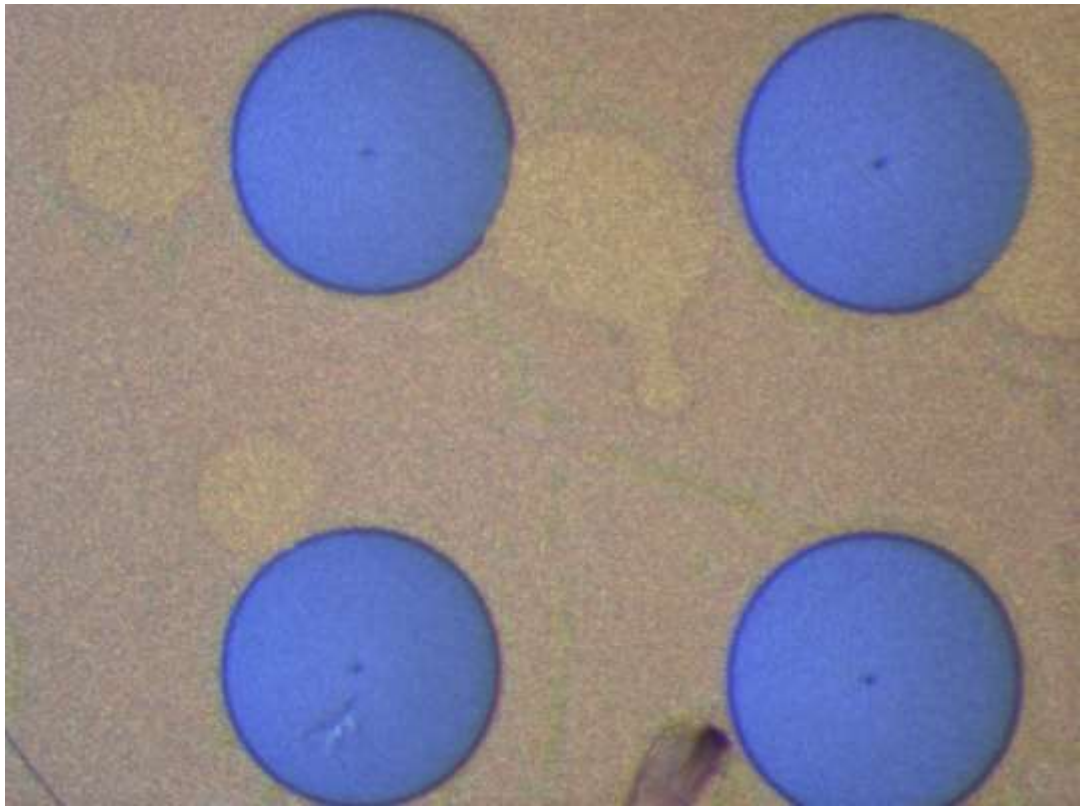
Optical microscope inspection



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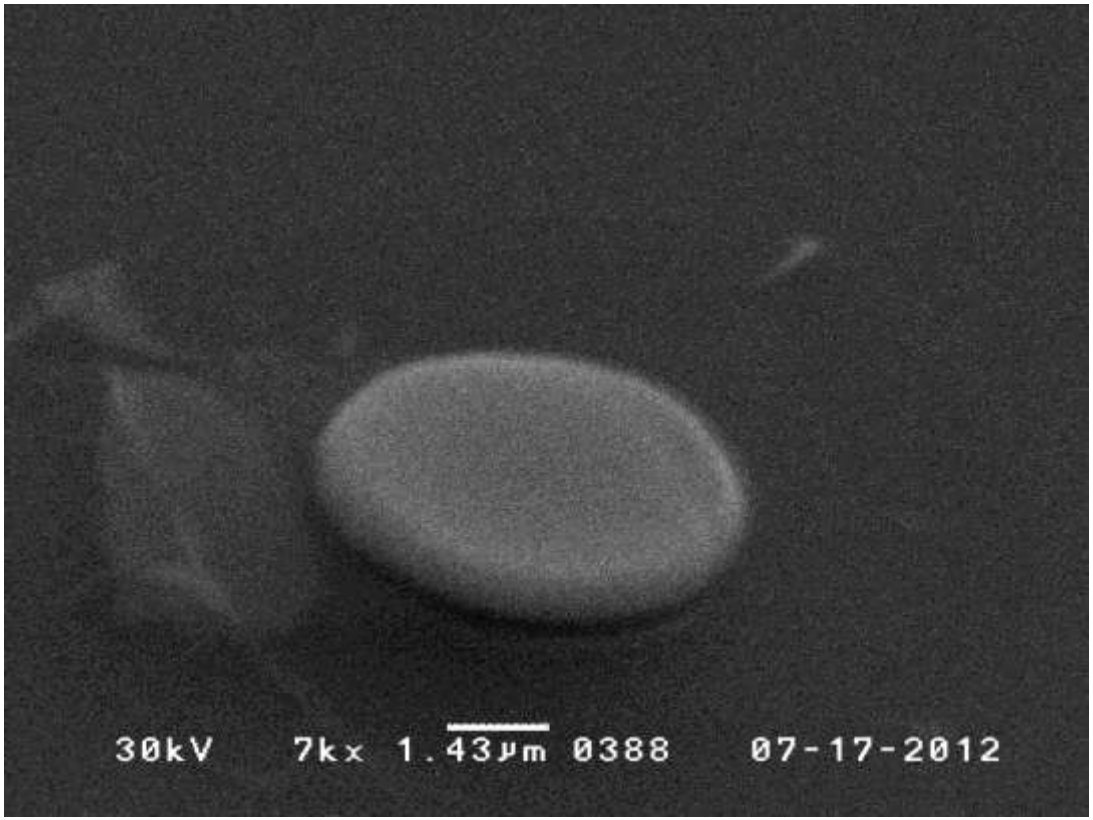
Scanning electron microscope inspection



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Conclusions

- MEMS-based fabrication processes have been used to successfully manufacture micro-dot laser targets
  - Good definition as individual targets
  - Good repeatability when used in high rep-rate arrays.
- Similar processes can also be used to fabricate other micron-scale structures such as backlighters, diagnostic filters and much more.

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THANK YOU

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IFE TARGET FABRICATION

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HiPER

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Thank You

C Spindloe, STFC,UK
P Hiscock, STFC, UK
N Sykes, Micronanics, UK
M Beardsley, STFC, UK
W Nazarov, St Andrews, UK
I Sari, U Southampton, UK
M Kraft, U Southampton, UK
G Schaumann, TUD, Germany
F ben Said, CEA, France
O Legaie, CEA, France
M Brookes, AWE, UK
D Wyatt, STFC, UK
S Serra, STFC, UK
D Haddock, STFC, UK
J Jiang, U Huddersfield, UK
R Leach, NPL, UK

J-P Perin, CEA, France
D Chatain, CEA, France
N Alexander, GA, US
E Koresheva, LPI, Russia
J.M Perlado, UPM, Spain
S Cuesta Lopez, UPM, Spain
G Schurtz, CELIA, France
D Guillaume, CEA, France
I East, STFC, UK
G Arthur, Scitech Precision, UK
F Hall, Scitech Precision, UK
D Barrow, U Cardiff, UK
D Harding, LLE, US

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Overview

- IFE Targetry Requirements
- Shell target mass production
- Complex target mass production
- Target Factory
- Is it achievable?
- Summary

IFE TARGETRY:

REQUIREMENTS

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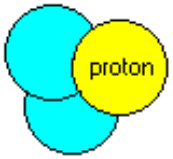
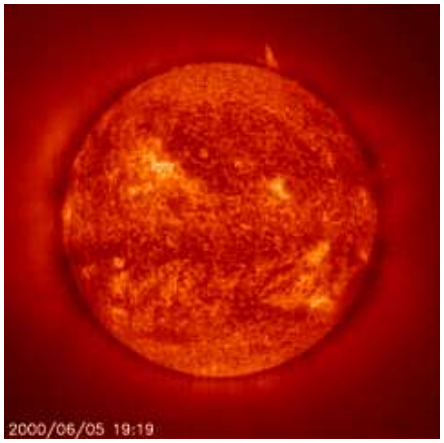
Fusion: Nature's energy solution

Deuterium molecule



Tritium molecule

The Sun: a natural fusion reactor



Core ~ 15 MK
~ 150 g/cc

$$D + T \rightarrow He + n$$

3.5 + 14.1 MeV

This energy is ~ a million times greater than in chemical reactions

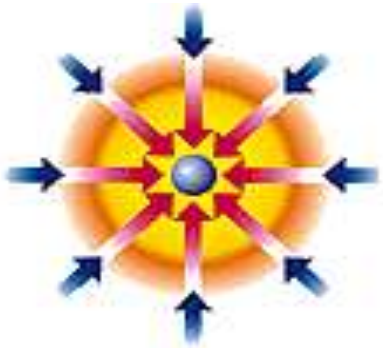
$$E = m c^2$$

Laser (Inertial) Fusion

A spherical, pulsed rocket

Lasers or X-rays symmetrically irradiate pellet

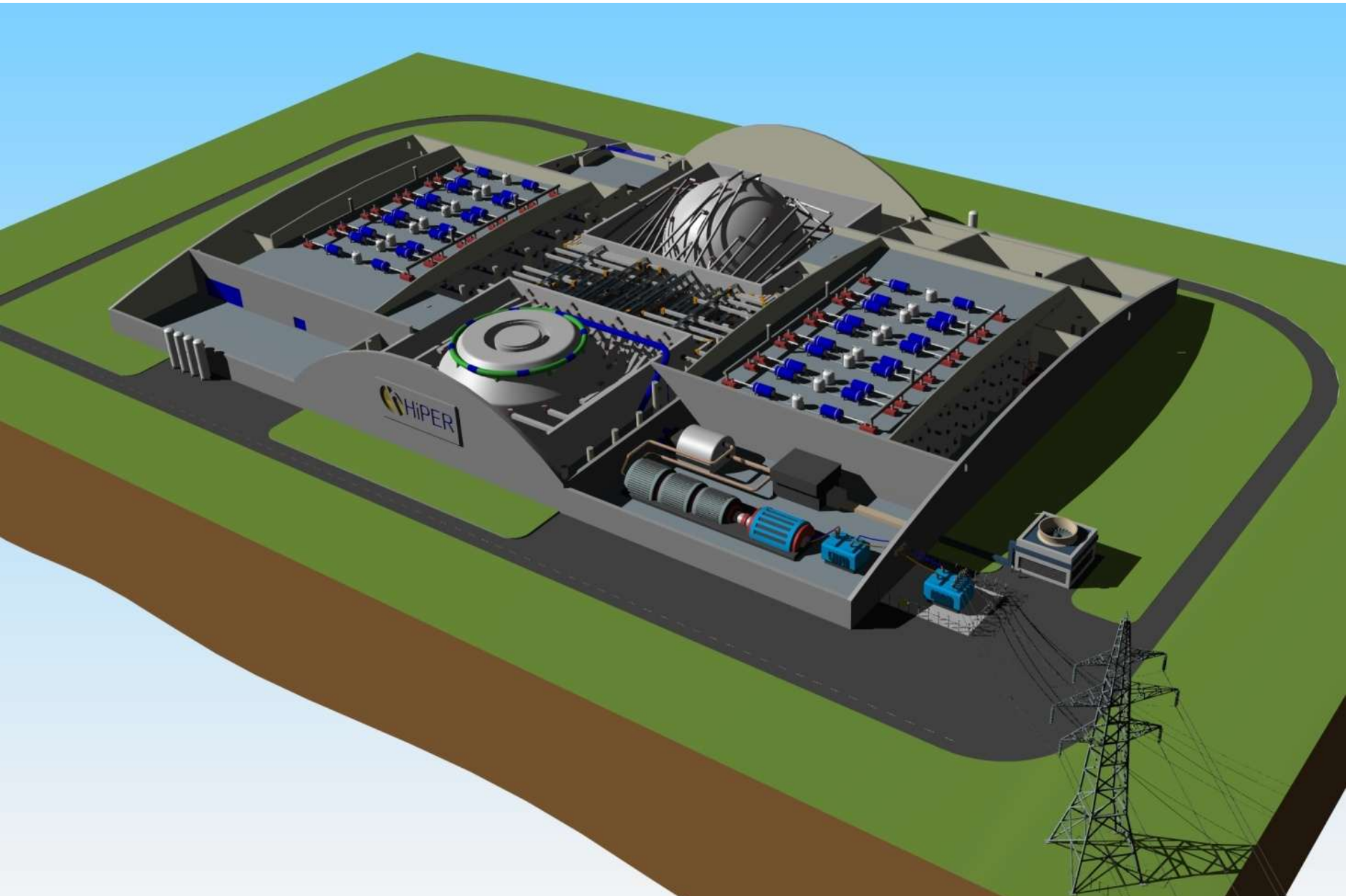
Hot plasma expands into vacuum causing shell to implode with high velocity



Material is compressed to ~1000 gcm^{-3}

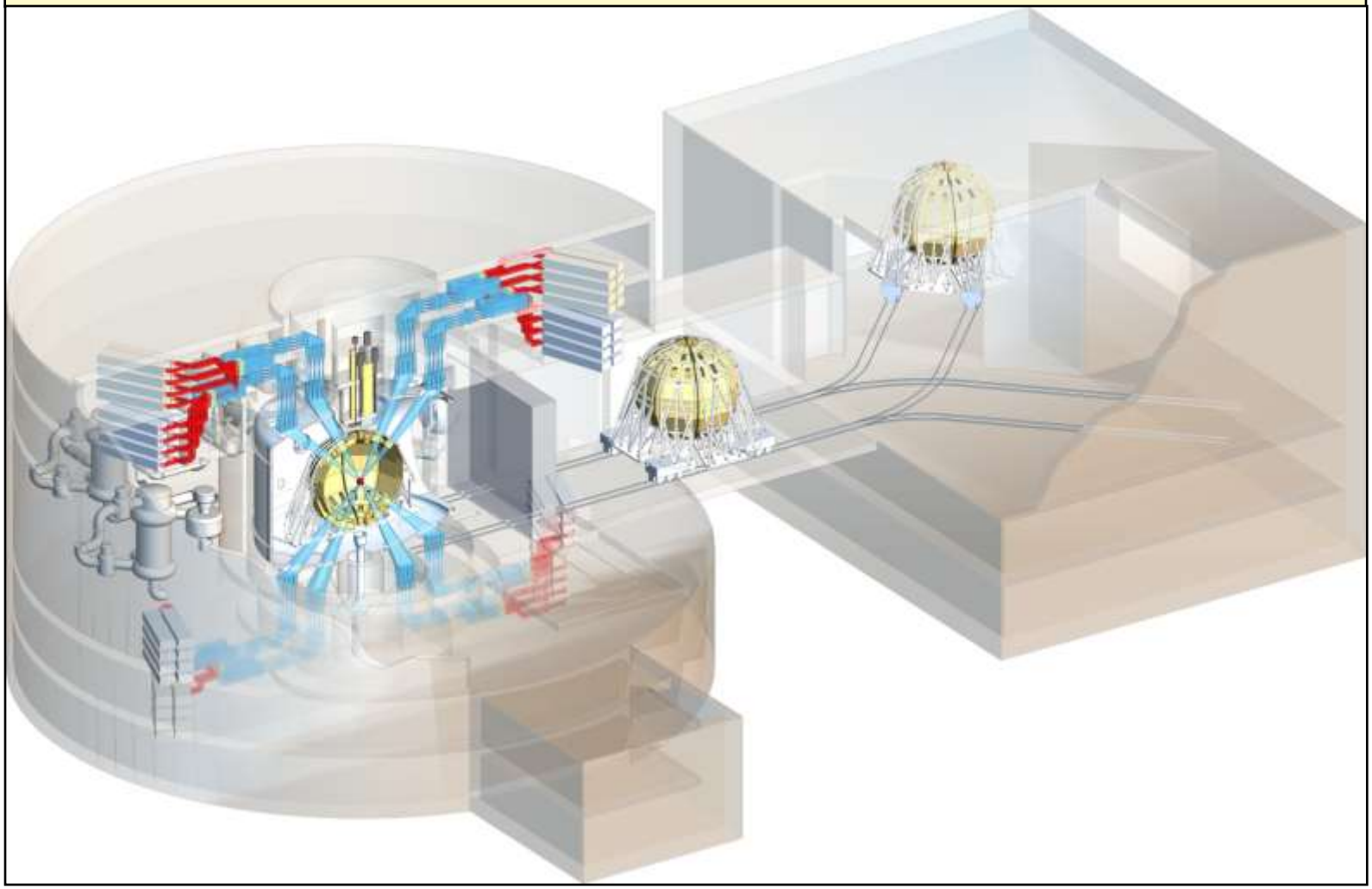
Hot spark formed at the centre of the fuel by convergence of accurately timed shock waves

Latest Facility Design



The chamber system can be transported for maintenance or replacement

Unsealed chamber, separate from the vacuum and optical systems



IFE Target Production Numbers

In continuous operation at 10Hz one IFE reactor will require:

~ 860 000 fuel capsules per day

In continuous operation at 16Hz one IFE reactor will require:

~ 1 380 000 fuel capsules per day

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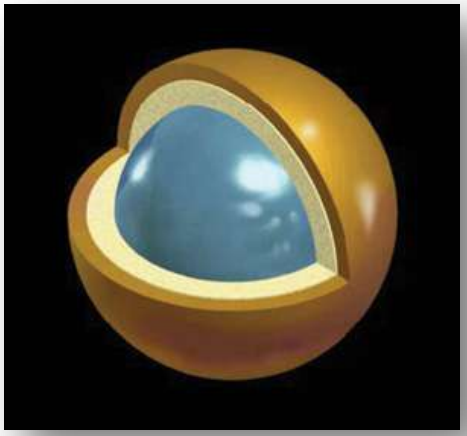
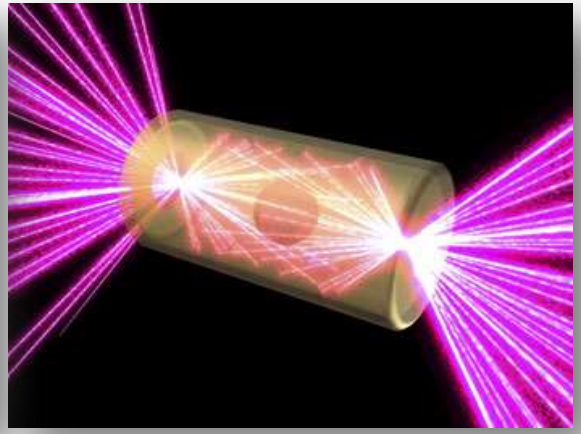
IFE Target Types

Three main generic types currently being considered. Many shared microfabrication challenges.

1. Shock Ignition Targets – DT filled shell targets with a low Z ablator on the outside
2. Indirect drive targets – DT filled shell with ablator in a hohlraum.
3. Fast Ignition – DT filled shell with ablator (and re-entrant cone)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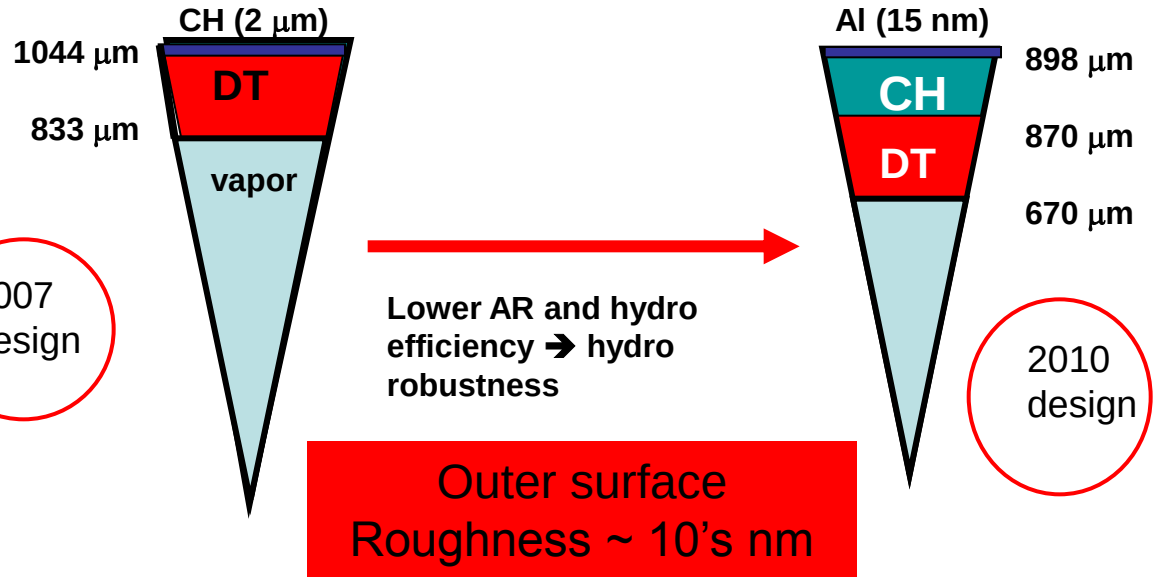
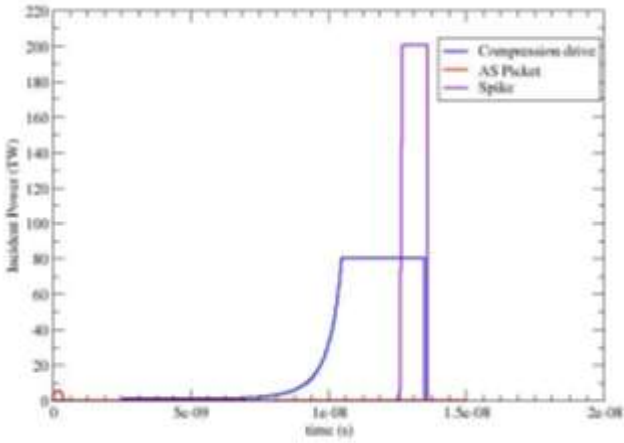
Shock Ignition Baseline Targets





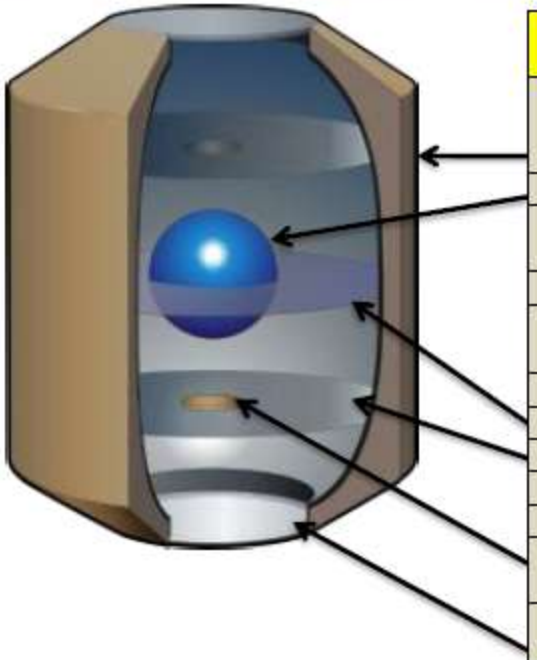
Outer surface Roughness ~ 10's nm

Inner ice Roughness ~ 1 μm

	IFAR 75%r_0	Mass	Compression	Vimplo	η %	Spike	Gain
ALL DT	4.5 (t=0) 30 (75%r_0)	.59 mg .29 fuel	180 kJ 50 TW 600 g/cc 1.5 g/cm^2	280 km/s	9%	160 TW 80 kJ	Y = 20 MJ G ~ 76
CH	3.4 (t=0) 18 (75% r_0)	.67 mg .38 mg fuel	260 kJ 80 TW 720 g/cc 1.83 g/cm2	240 km/s	5%	200 TW 150 KJ	Y = 32 MJ G ~ 80

G Schurtz et al

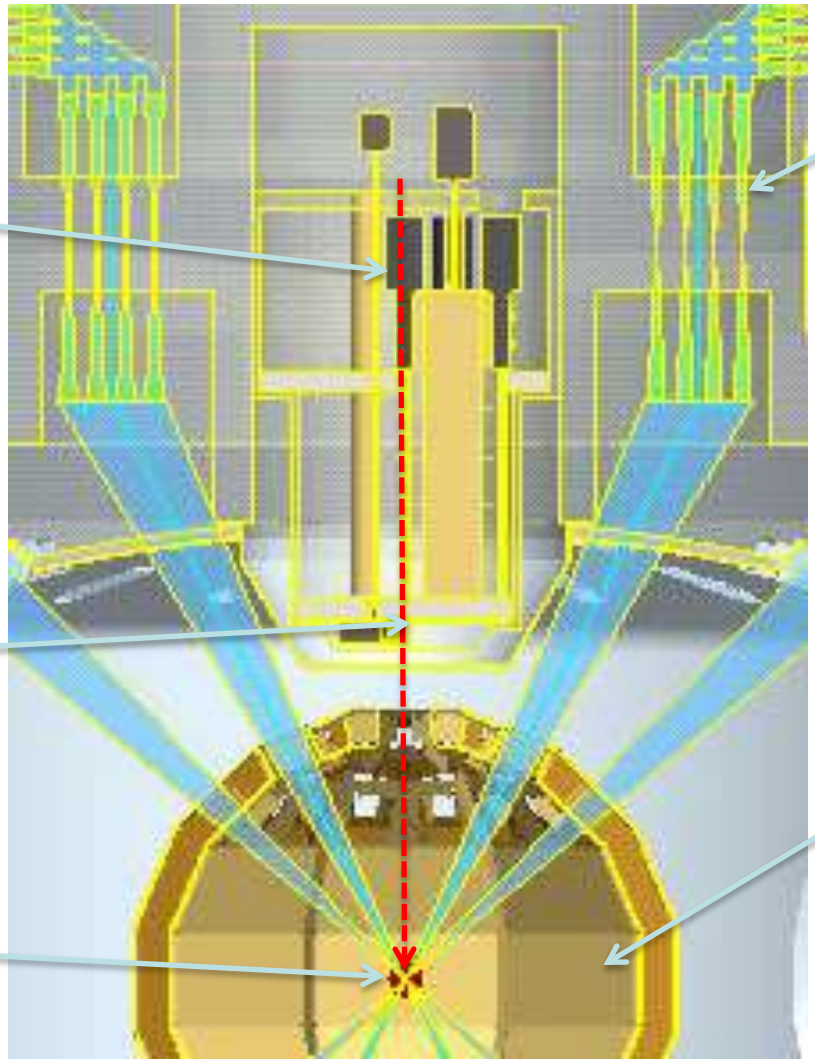
LIFE exhaust characterization is essential for design of tritium systems



Component	Dimensions	Material	~Mass (mg)	Alternate materials
Hohlraum	Length = 1.54 cm ID = 0.89 cm Thickness = 0.05 cm	Pb; 5% Sn or Sb	2579	High-Z
Capsule				
Ablator	OD = 0.382 cm Thickness = 75 um	C	11.6	CH;Be,B
Dopant		Ta	0.0021	Ge
Foam	Thickness = 142 um	DCPD (< 20 mg/cc)	0.1389	SiO_2 (5 mg/cc)
DT	Thickness = 142 um	DT	1.3889	
Support	Thickness = 110 nm	C	0.0488	polyimide
IR window				
Substrate	Thickness = 400 um	C	0.2439	polyimide
Metalization	Thickness = 30 nm	Al	1.01E-05	Ag,Au
P2 shield	OD = 0.191 cm Thickness = 10 um	Pb; 5% Sn or Sb	6.50	High-Z
LEH window	Diameter = 0.45 cm Thickness = 500 um	C	0.2439	polyimide
Total mass			2599	

- Strong interface between target material choices and design of T systems
- Optimized LIFE target design eliminates CH substrate (no protium)
- Tritium per target < 1 mg (~ content in one EXIT sign)
- 0.5% chamber clearing ratio with partial gas recirculation facilitates T recovery

Fusion targets are injected at high acceleration rates into a hot chamber



The target must arrive at chamber center in the correct configuration for ignition

IFE Targetry – Key Challenges

Target technology is one of the key challenges for IFE

The main IFE Target Fabrication challenges

- **Scaling of processing and materials to micro-domain**
- **Industrial scaling for mass production**
- **Novel production processes and materials**
- **Tritium handling**
- **Fine control cryogenics/layering**
- **And all at ~ 25 – 30 €c per target !**

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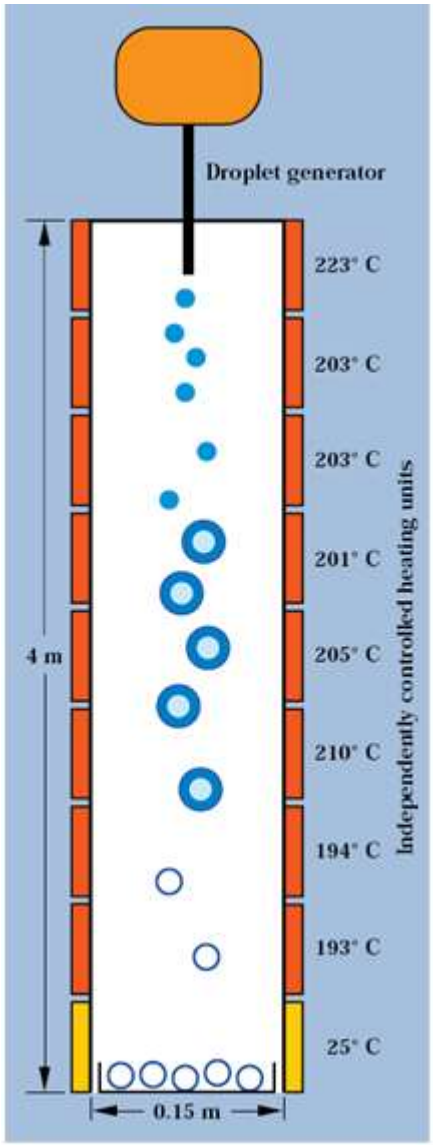
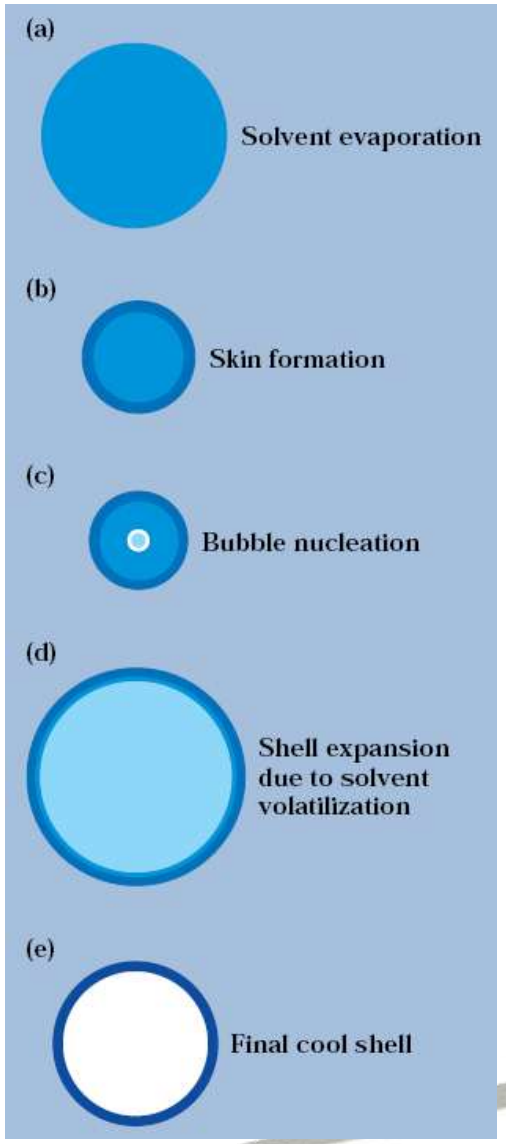
SHELL (MASS) PRODUCTION

Science & Technology Facilities Council

1. Drop Tower





1 - A double orifice generator injects a droplet of polymer solution into the tower.

2 - A polymer membrane skin forms at the surface due to solvent evaporation.

3 - When the droplet temperature exceeds its boiling point the shell inflates.

4 - The final size and quality will depend on the cooling rate and symmetry of the hot polymer shell.

Requires expensive equipment.

Robert Cook, Creating Microsphere Targets for Inertial Confinement Fusion Experiments, Lawrence Livermore National Laboratory.

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2. Microencapsulation: Double emulsion method

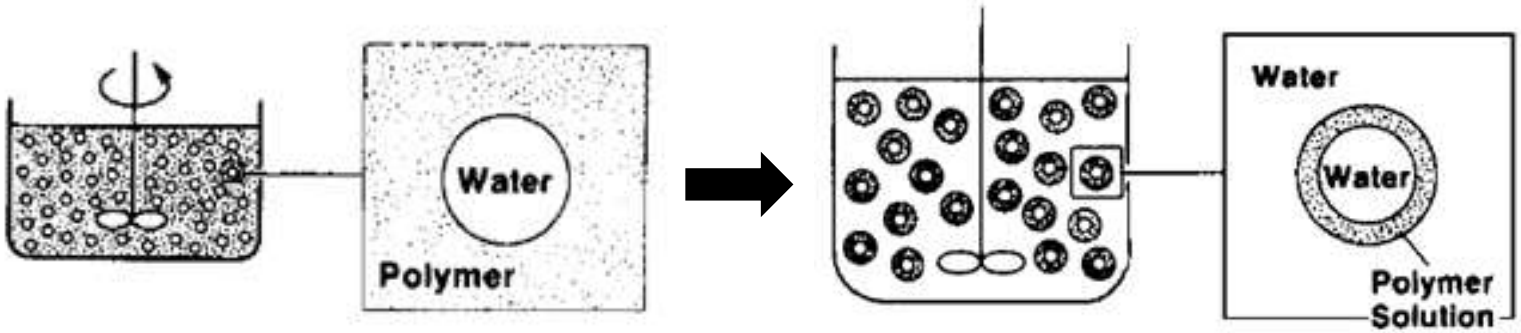
1 – Emulsify water into polymer solution (oil phase) with surfactants and mechanical agitation.

2 – Pour emulsion into second water phase while maintaining mechanical agitation

3 – Evaporate polymer solvent

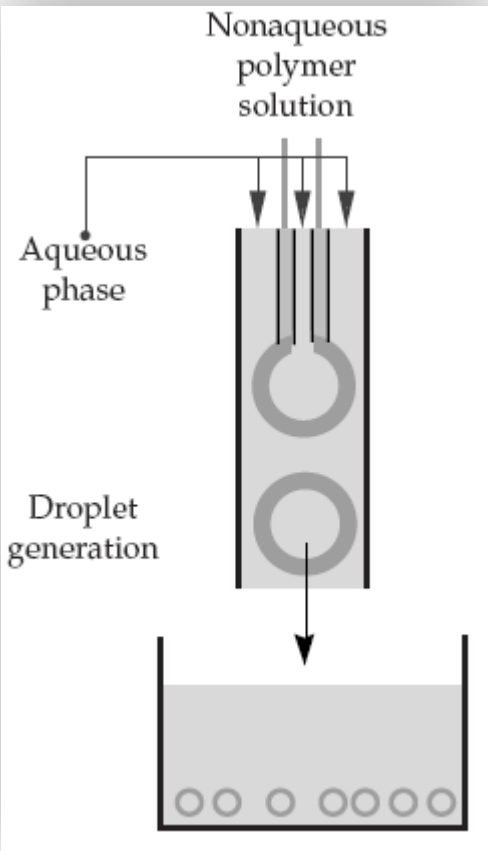
4 – Remove microshells from second water phase

5 – Evaporate water from inside microshell



Guillermo Velarde, Yigal Ronen, José María Martínez-Val, Nuclear fusion by inertial confinement: a comprehensive treatise.

2. Microencapsulation: Triple orifice droplet generator



To form W/O/W emulsion droplets with reasonably constant diameters.

The smallest orifice flows water to the inner phase.

Around it, the second orifice flows the polymer solution.

The third orifice around it dispenses water and surfactant solution.

The droplets are dripped into a stirred beaker of water.

Adjusting the flow rates of the various solutions changes the outer diameter and shell thickness.

R. Cook, The development of plastic targets for NIF targets, *ICF Semiannual Report October 1999—March 2000, Volume 1, Number 1, LLNL*

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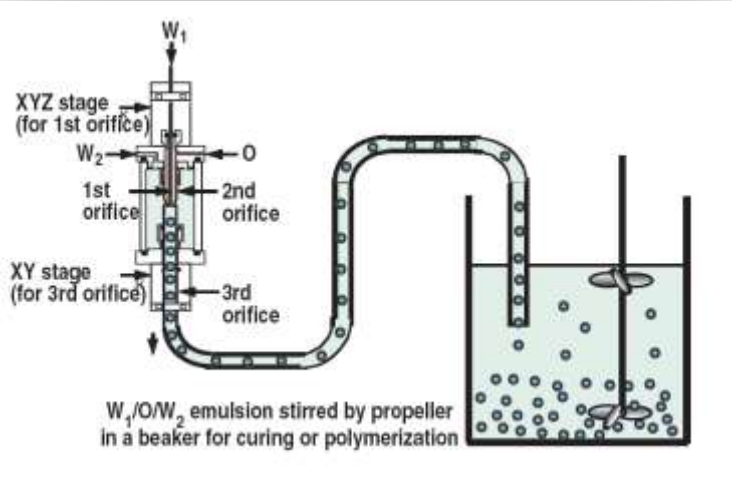
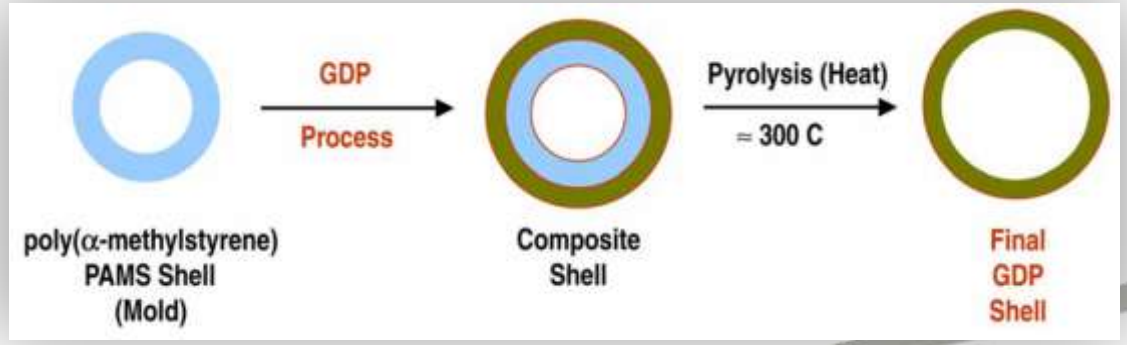
2. Microencapsulation: Depolymerisable Mandrel Technique

PAMS (Poly-α-methyl–styrene) mandrels are produced using a microencapsulation process.



W1 Water
O 11%wt PAMS in Fluorobenzene
W2 PVA solution 0.2%wt

When dried the PAMS mandrels are over coated with a Glow Discharge Polymer (GDP) to the desired thickness.



Evelyn M. Fearon, Adapting The Decomposable Mandrel Technique To Build Specialty ICF Targets, 11th Target Fabrication Specialists' Meeting Orcas Island, Washington September 8–12, 1996.

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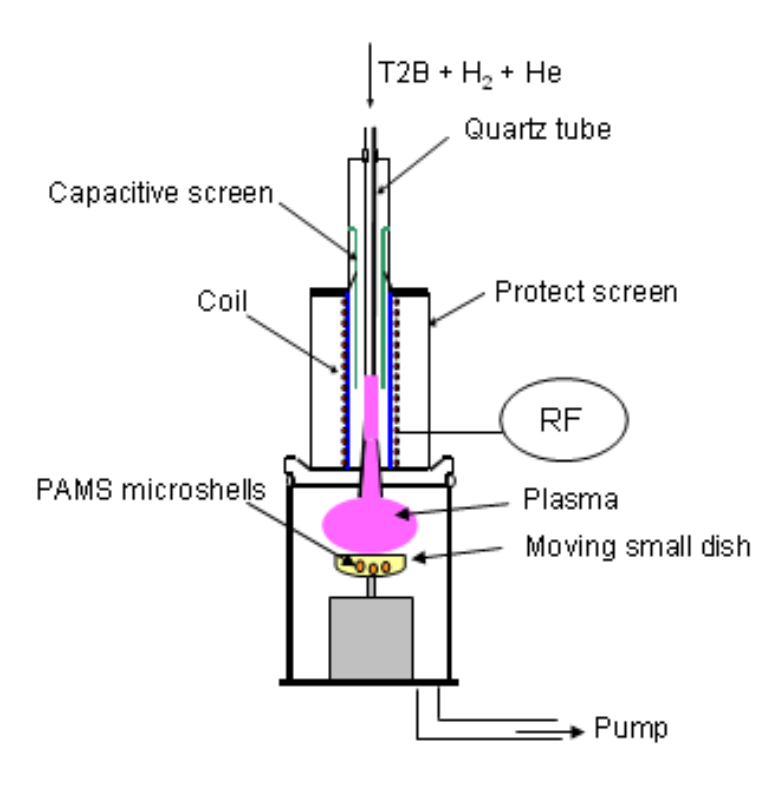
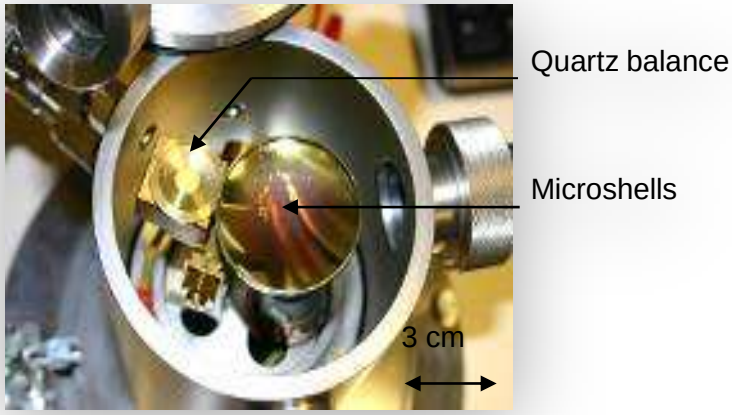
Microballoon PAMS





Discharge polymerization coating schematic (left) and deposition chamber (right)

F. Bensaid
(CEA-Valduc-DRMN-L.M.C)

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SHELL (MASS PRODUCTION) FILL AND CRYOGENIC LAYERING

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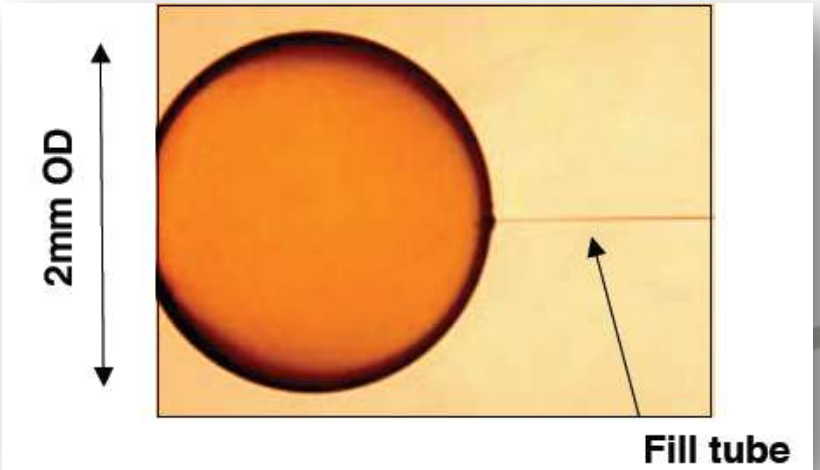
D-T Filling

Filling of the shell with D-T fuel can be either carried out using a diffusion fill or where the ablator layer prevents this by drilling a small hole attaching a fill tube and introducing the cryogenic liquid through the tube.

1) Diffusion Fill
   DT gas is permeated through the wall in a controlled manner to prevent buckling of the wall in a high pressure cell. The cell is then cooled to ~20K to condense the gas.

2) Fill Tube
   A small hole is laser drilled about 5μm in diameter and a tube is bonded into the shell. The shell is then cooled close to the triple point of the liquid and then the tube is used to introduce the fuel.



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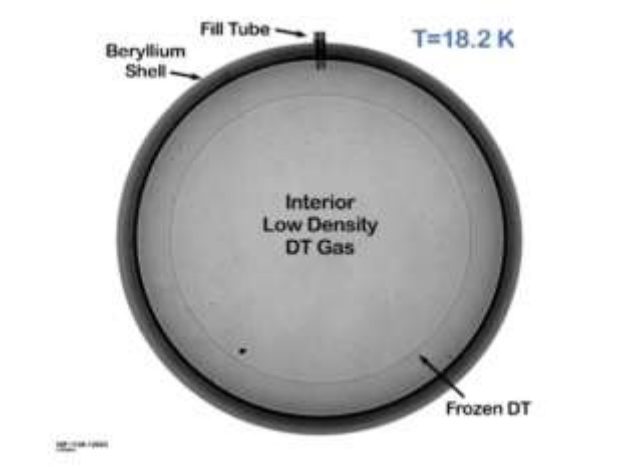
Layering

1) If enough tritium is present in the shell can use **Beta layering**.

Nuclear decay of the tritium produces a beta particle that loses its energy to heat causing most redistribution from regions where layer is locally thickest.

2) If there is not sufficient tritium (e.g. H-D-T target) a **layering chamber** is used.

Filled shells are held in a cryogenic environment and repeatedly slightly heated (with an IR laser) and then cooled causing redistribution.



NIF capsule showing DT ice layer

Both layering processes typically take ~24 hours. Scalability to IFE?

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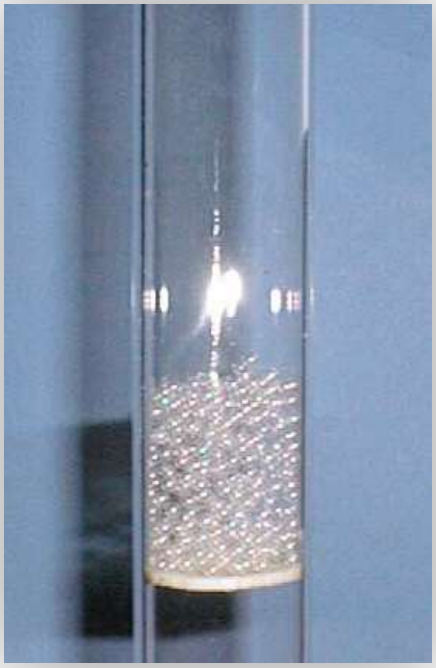
Fluidized Bed Technique

General Atomics (and Shafer) have proposed a fluidised bed technique for mass production layering.

- Possible issues with surface damage.

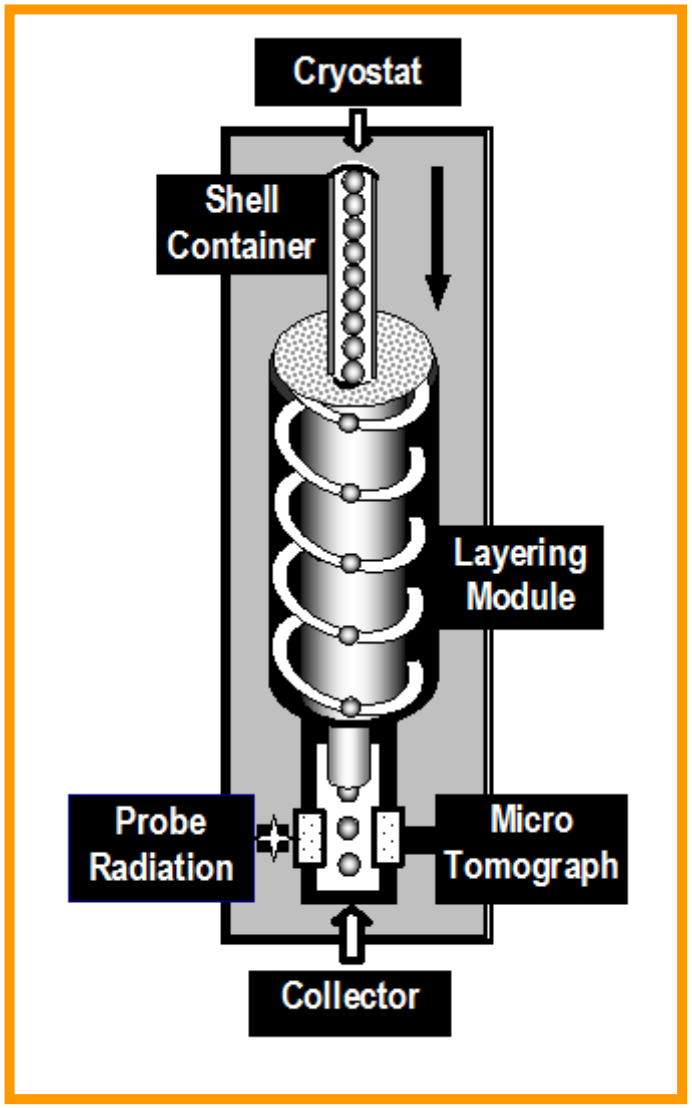
- Multiple nucleation sites so possible nanocrystallinity



Capsules layering in a fluidized bed

N Alexander, Report GA-A23813

LPI: D-T Fill and Layering



Layering Mechanisms – Free Standing Target (FST) Layering

Permeation fill of the shells in a cryostat.

Rolling of the shells down a layering channel

Characterisation of the layer at the end.

Issues

Fuel needs to be a single crystal layer?

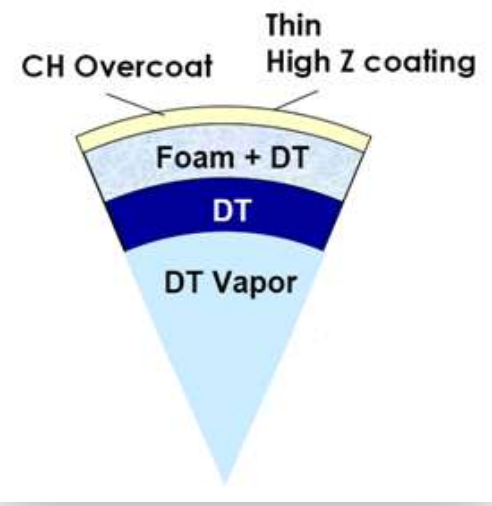
FST filling may give polycrystalline DT

- I.V.Aleksandrova, E.R.Koresheva, I.E.Osipov, *et al. Free-Standing Target Technologies for ICF.* 2000 Fusion Technology 38 No1 p.166
- I.V.Aleksandrova, S.V.Bazdenkov, V.I.Chtcherbakov et al. *An efficient method of fuel ice formation in moving free standing ICF/IFE targets.* J.Phys.D: Appl.Phys. **37**, 1-16, 2004

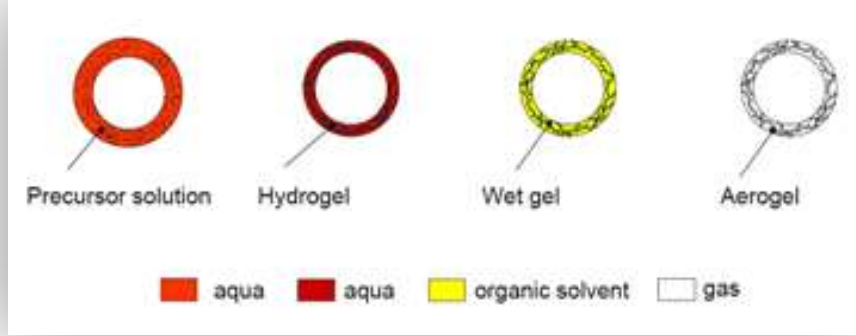
Don’t Layer – Use Foam Shells

Target designs including foam shells have been proposed for ICF. Potential foam materials include polystyrene (PS), resorcinol-formaldehyde (RF) and divinylbenzene (DVB).

With a foam layer the DT fuel may wick into the foam thereby reducing/removing the issues associated with layering.



Laser plasma interactions 5: Inertial confinement fusion, M.B. Hooper.



Keiji Nagai, et al., Foam materials for cryogenic targets of fast ignition realization experiment (FIREX), Nuclear Fusion 45 (2005) 1277–1283.

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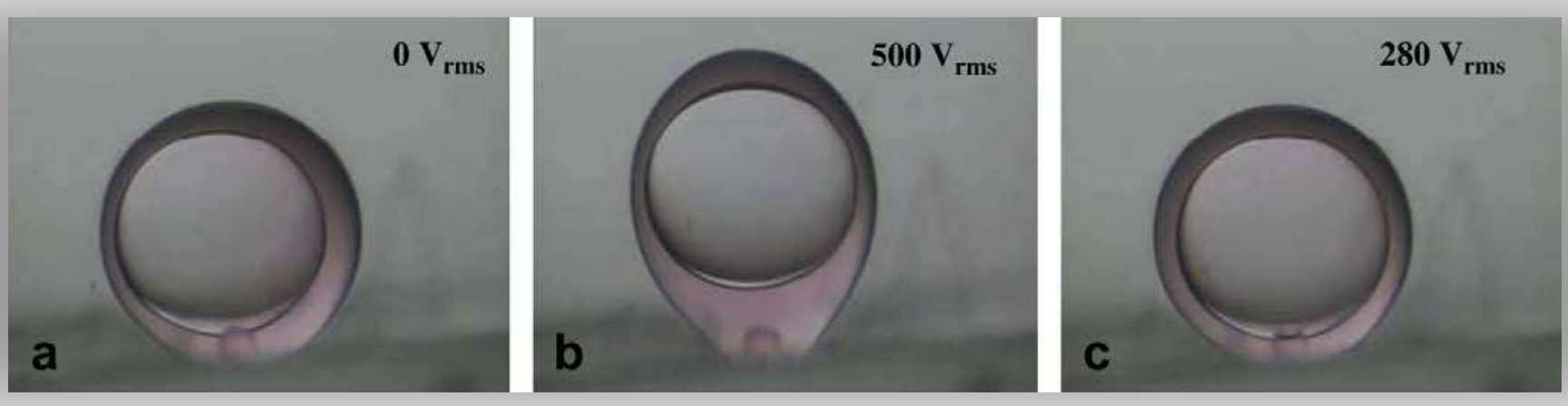
Advanced Techniques: Microfluidics

- Harding* has proposed a six phase programme to produce layered shells based on microfluidics.



* 1) W. Wand et al, On-Chip Double Emulsion Droplet Assembly Using Electrowetting-On-Dielectric and Dielectrophoresis, Fus. Sci. and Tech, **59,1**, pp 240-249

2) Z.-M. Bei, et al., Forming concentric double-emulsion droplets using electric fields, Journal of Electrostatics (2009), doi:10.1016/j.elstat.2008.12.013

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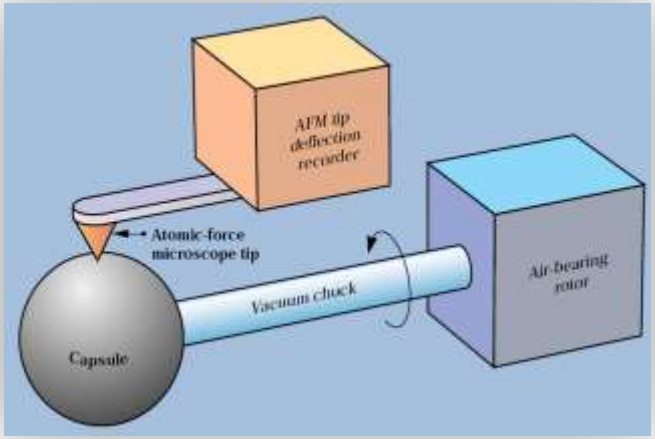
Characterization Methods

Shells can be characterised for the outside wall roughness using an AFM.

Robert Cook, Creating Microsphere Targets for Inertial Confinement Fusion Experiments, Lawrence Livermore National Laboratory.



A. Iwamoto, K. Nagai, Development of the Foam Cryogenic Target for the FIREX Project, IF/P5-1

Optical interferometry /shadowgraphy used to characterize wall thickness and defects.

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HIGH ASPECT RATIO MICROCOMPONENT/ MICROTARGET MASS PRODUCTION

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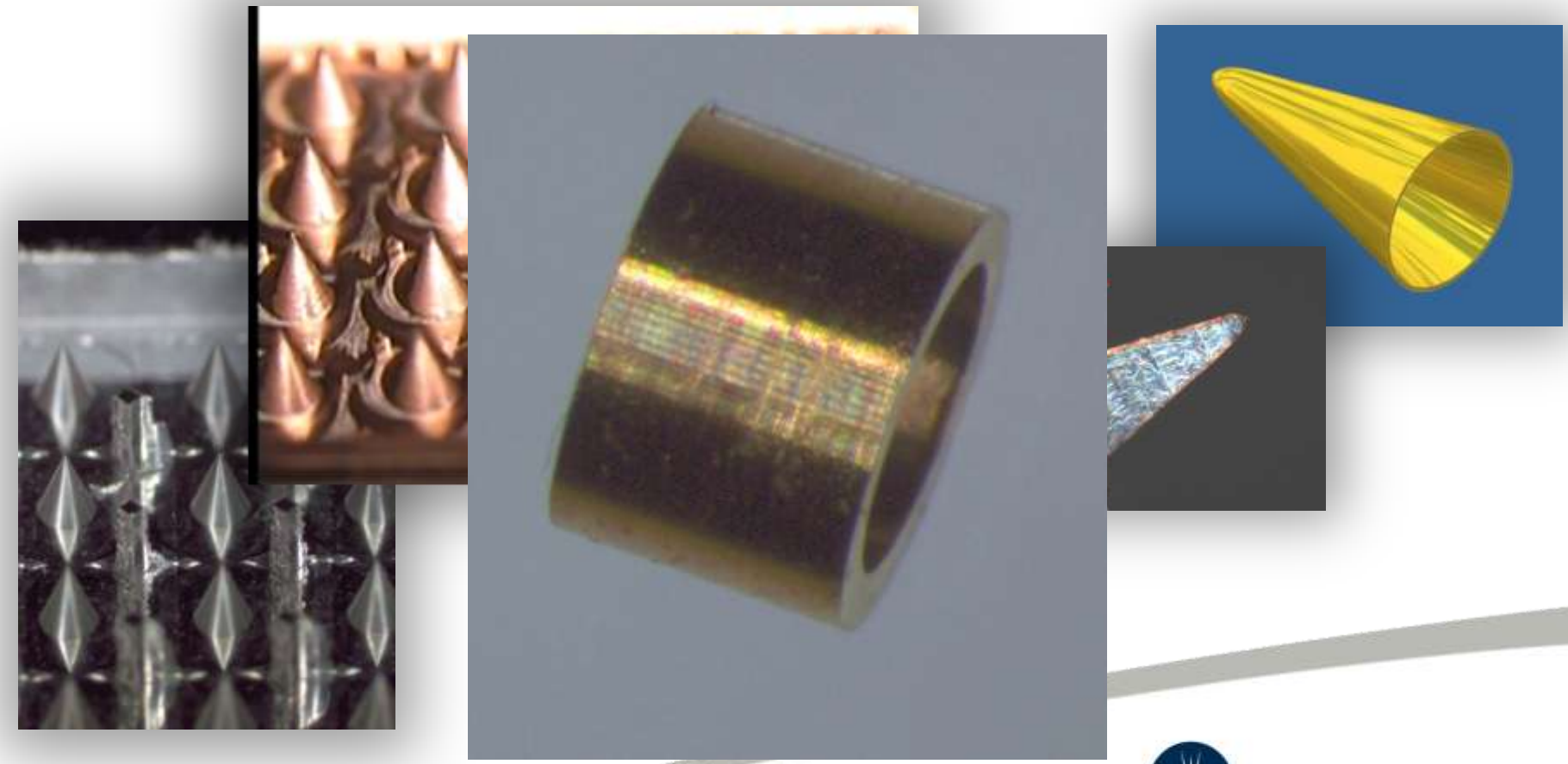
High Aspect Ratio Microcomponent Mass Production 1) Micromachining

Significant progress in production of 3D microcomponents



Show target

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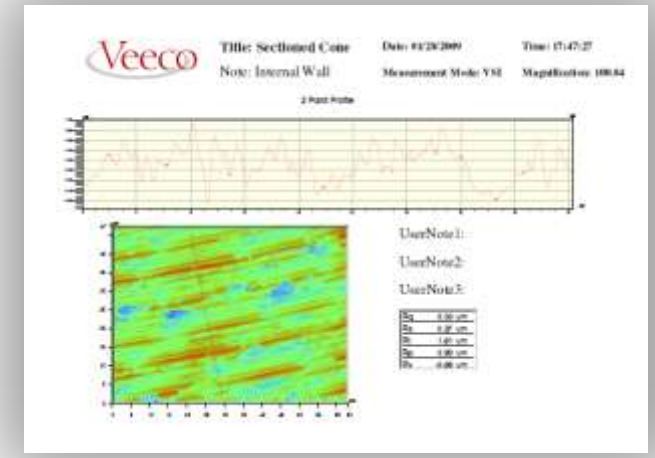
High Aspect Ratio Microcomponent Mass Production 1) Micromachining



Iteration between metrology and processing parameters is enabling improved quality, especially surface roughness. (Sub micron roughness achievable 0.25um Ra)

The specific CNC allows large area machining suitable for Pressing and Injection molding

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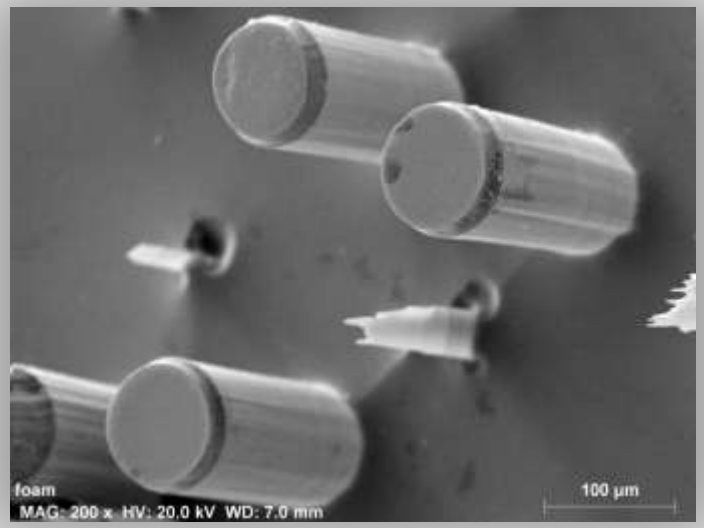
High Aspect Ratio Microcomponent Mass Production 2) MEMS

Advanced lithography techniques for production of high aspect ratio structures.



- Vertical side patterned halfraum production.
- Entire wafer processed in 1 lithography step

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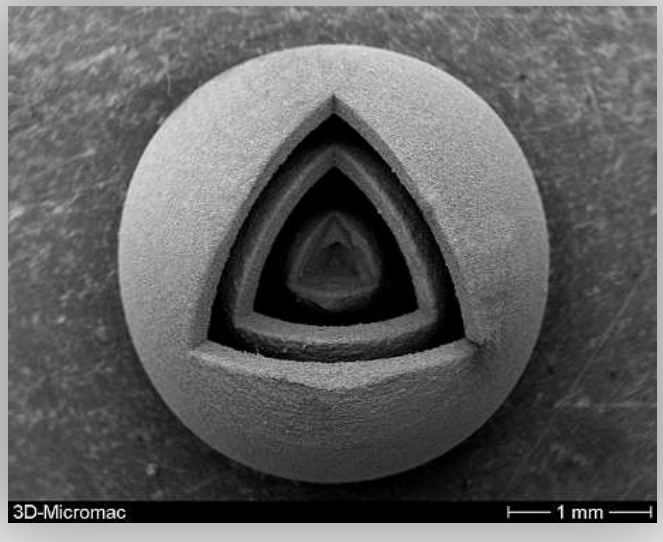
3) EmergingTechniques

1) ALM – Laser Sintering

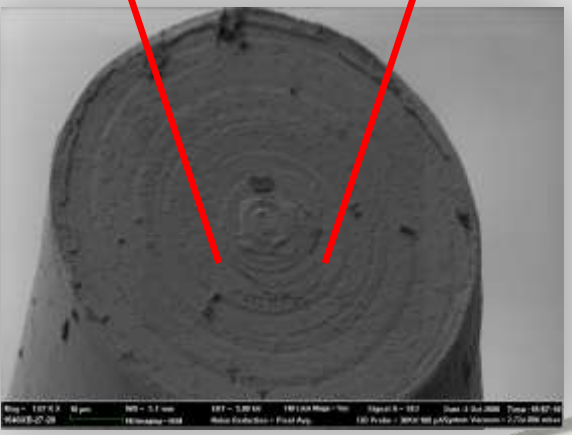


EOS Sphere (Mo) (Laser sintered)

2) Injection Moulding

3) Pressing

4) Focussed Ion Beam



5) Atomic Layer Deposition

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INJECTORS

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Facilities Council

Target (Injection) Survivability

"We calculated the stress in DT target over a range of injector acceleration values up to 10,000 m/s^2 for three target support methods; support on a hard flat surface, support within a cup in a low-density (50 mg/cm^3) foam, and on a flexible membrane.

Calculations showed that the DT would survive without yielding for the foam and membrane supports with 10,000 m/s^2 acceleration, but would not survive acceleration on the flat plate for any useful value of acceleration."

Information taken from report commissioned from General Atomics by HiPER:

Task 11.4.2: Modeling of Injector accelerated cryogenic layered targets and assessment of survivability

Task 11.4.3 Comparative analysis of injector designs (suitable for cryogenic targets injected into radiation environment)

Injector Requirements

The 2010 HiPER-EU tender (direct drive targets) included the following very demanding specifications **which cannot be met by any current injector technology**

The injector shall introduce targets into the chamber at a velocity of between 500m/s +/-10% to 1000m/s +/-10% and at a repetition rate of between 5 and 10Hz.

The time between target arrivals shall be between 100ms and 200ms +/- 2ms, (this is dependent on selected repetition rate).

The injector should be capable of passing targets through the chamber centre with an x,y,z accuracy of better than 10mm at chamber centre point.

Target Injector Types

TARGET INJECTORS

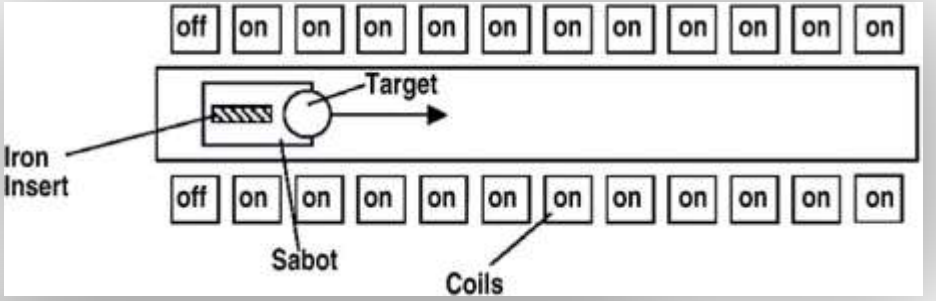
1. Mechanical Injectors

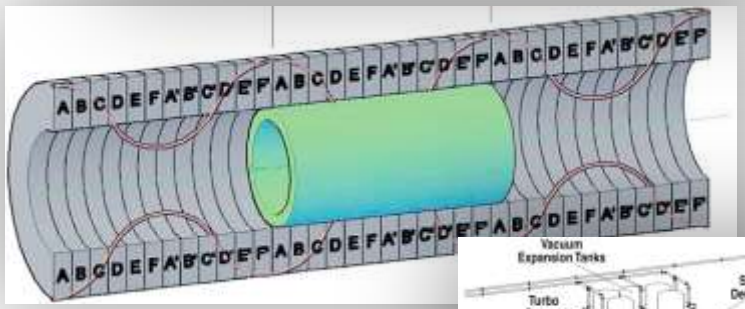
2. The Magnetic Accelerator

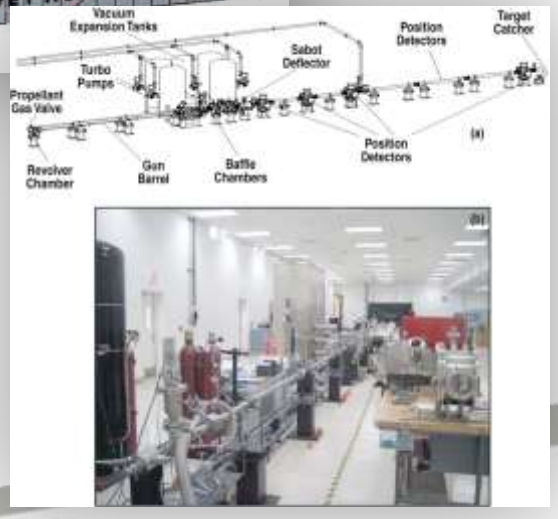
3. The Gas Gun

4. The Electrostatic Accelerator

5. Magnetic Slingshot







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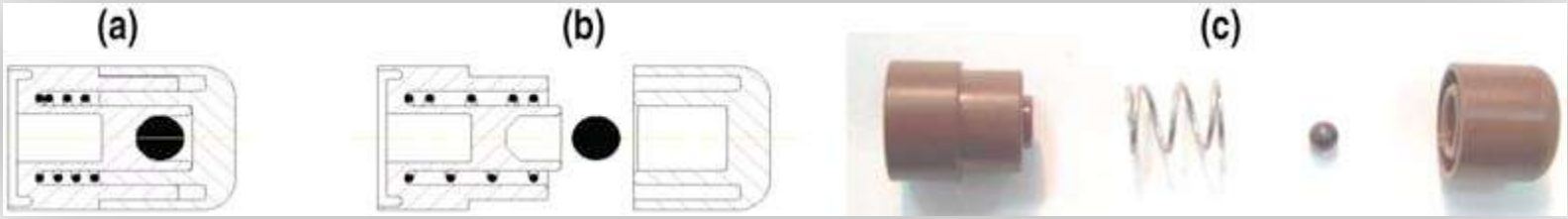
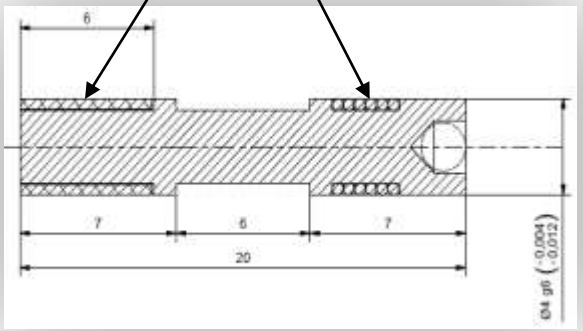
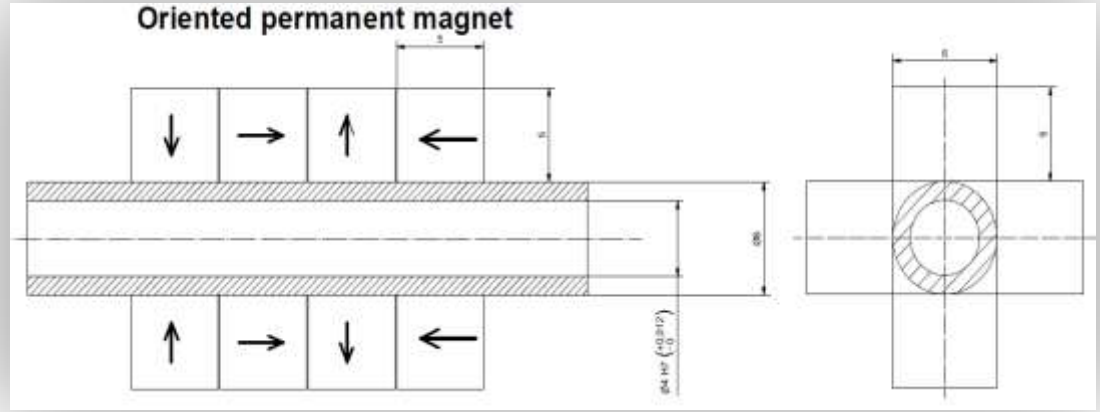
Sabots and Sleighs



Picture courtesy General Atomics

Stabilization coils





Magnetic bearing

EU Tracking Tender (Dec 2009)

4.1 Target Tracking System Parameters (All Targets) (*Extract*)

- Velocity Measurement +/-1μm/s over the range 400 to 1100m/s
- Positional measurement to within 1 μm x,y,z
- Positional Accuracy +/-1μm x,y,z
- Timing Accuracy +/-1ns

- Confident that tracking both during acceleration and in chamber will be achievable
- Therefore if the injector cannot achieve accuracy then a combination of either steering of target, or the optics will allow engagement of the target

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TARGET FACTORY PRODUCTION LINE

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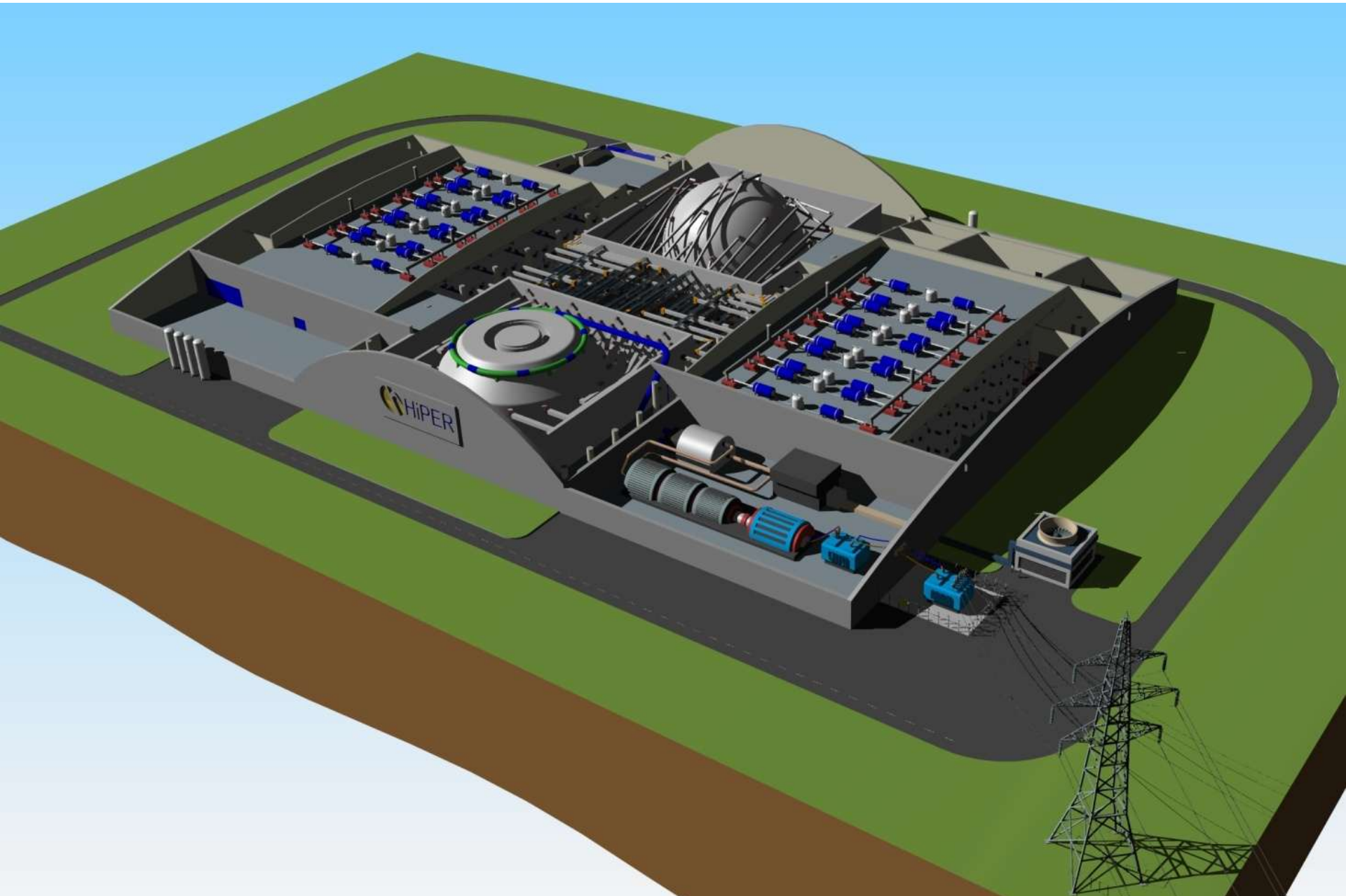
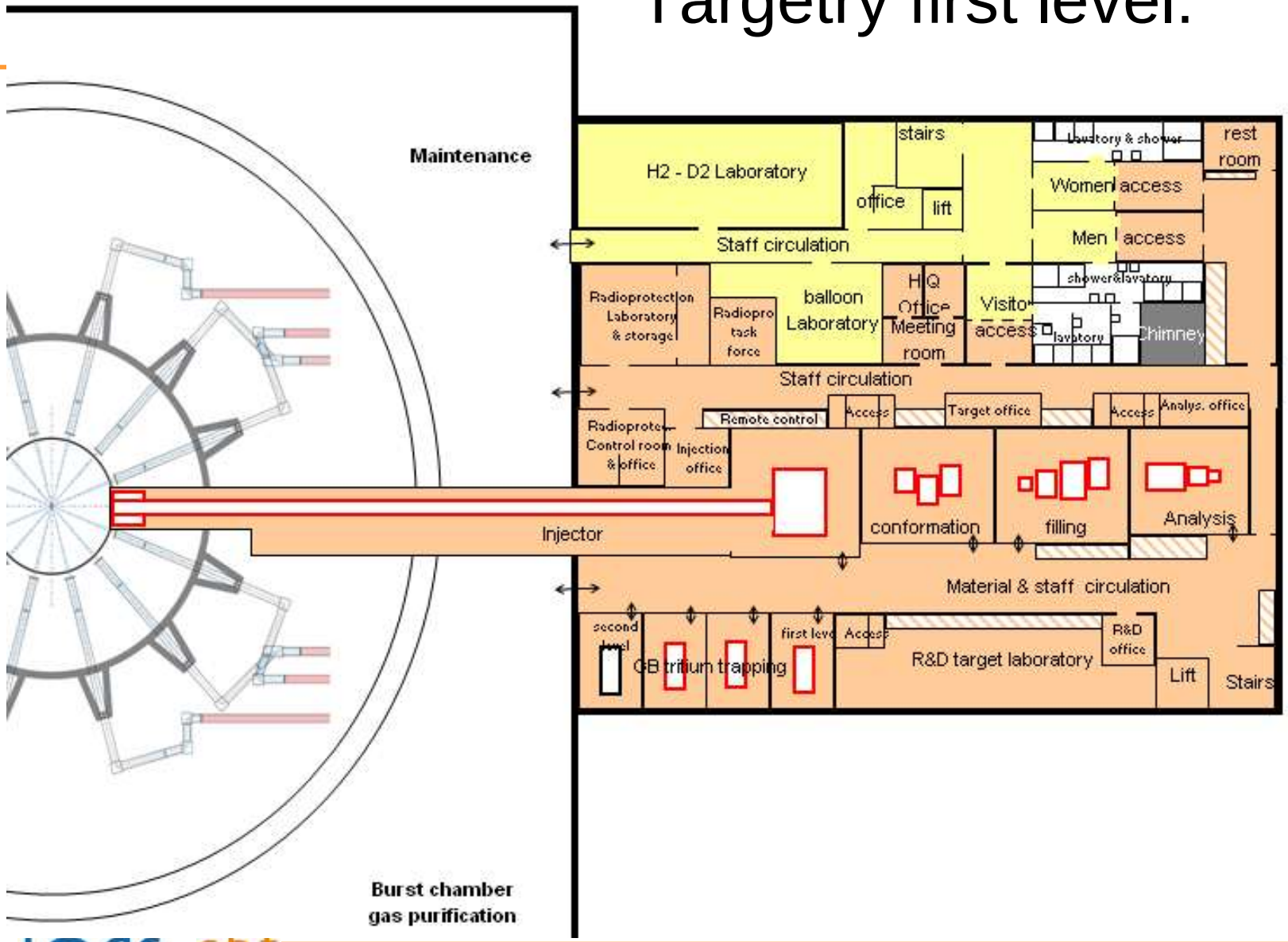
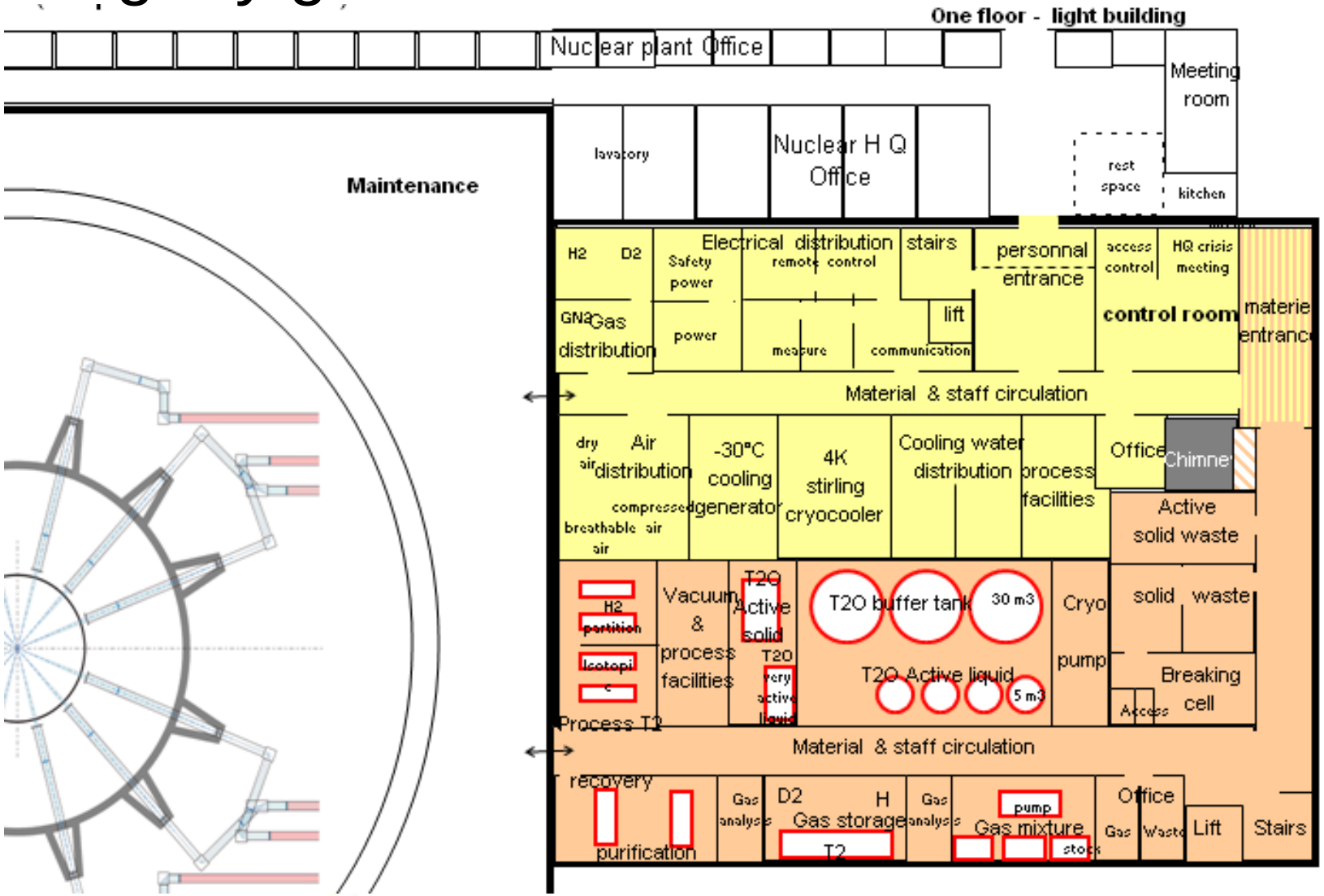
Latest Facility Design



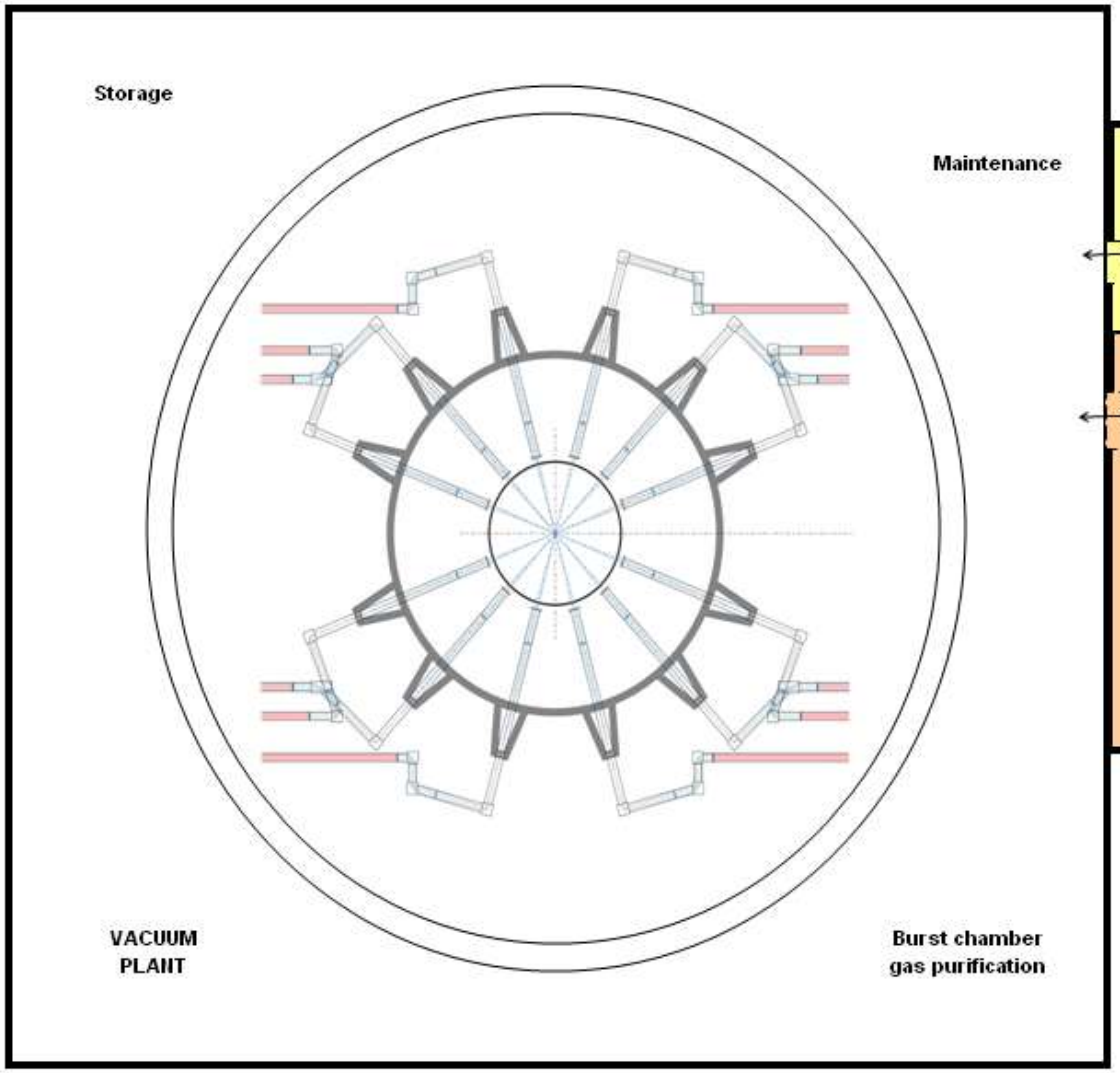
Targetry first level:

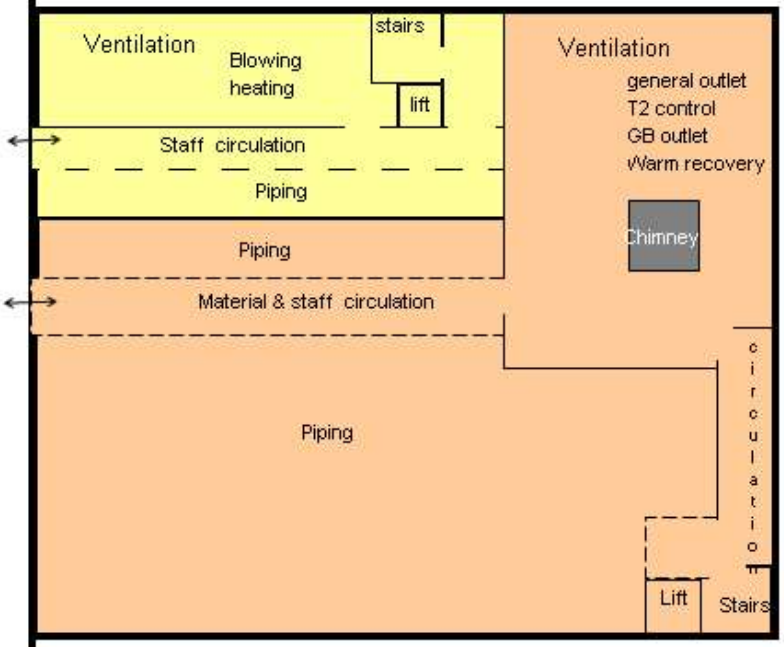


Targetry ground level:



Targetry second level:





Target Factory Operation: Integrating Advanced Target Technologies and Pragmatics of the Production Line

- Operating regime to be decided from economic analysis; six sigma aspiration, pragmatically 3-4 sigma.
- Sampling frequency and techniques to be decided.
- Highly automated with some ability for remote handling.
- On-line buffer period built in.
- Transfer technique: mechanical vs levitation.

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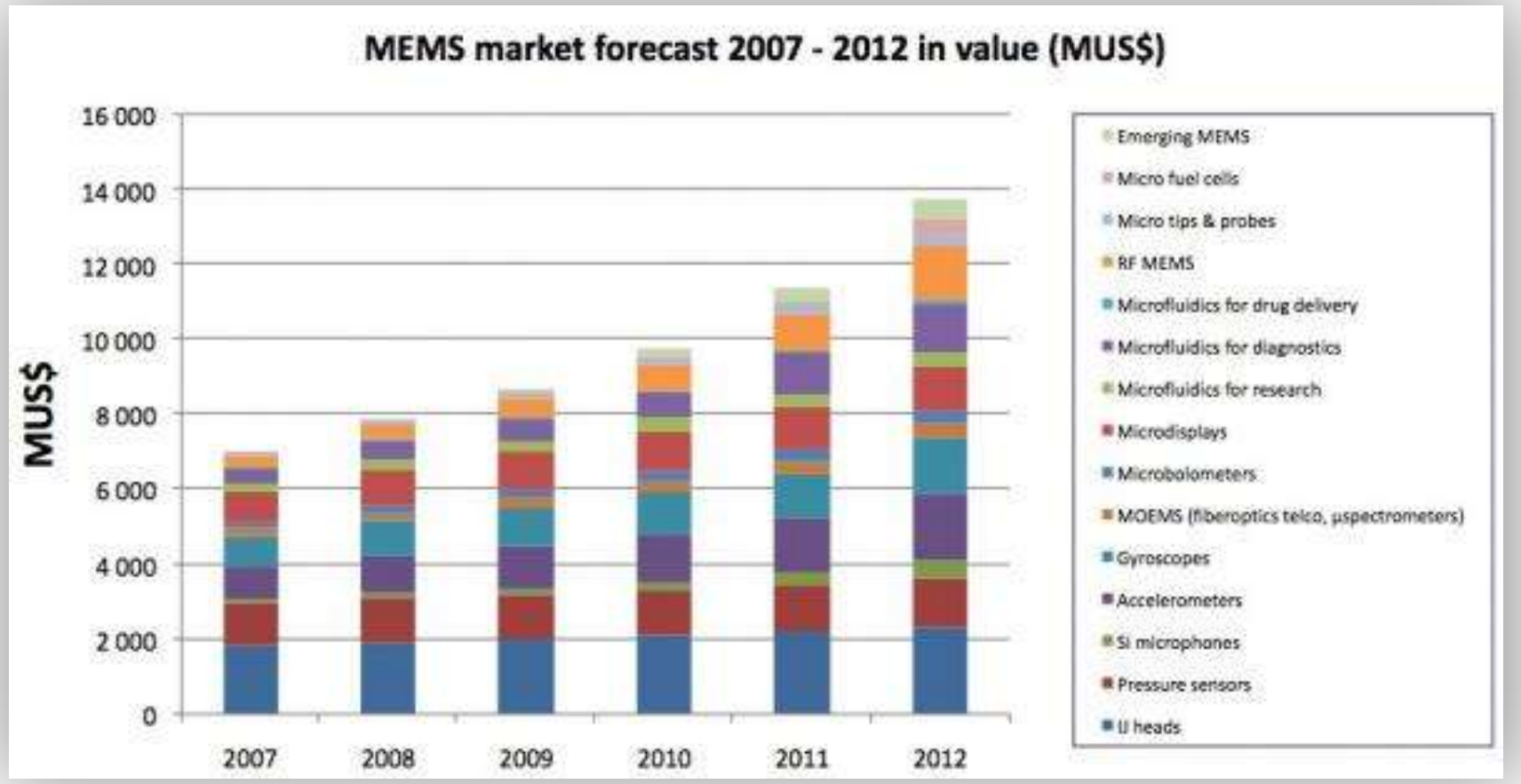
IS COST-EFFECTIVE IFE MICROTARGET PRODUCTION ACHIEVABLE?

Industrial Comparisons (1)



Yole 2007-2012 global MEMS market forecast

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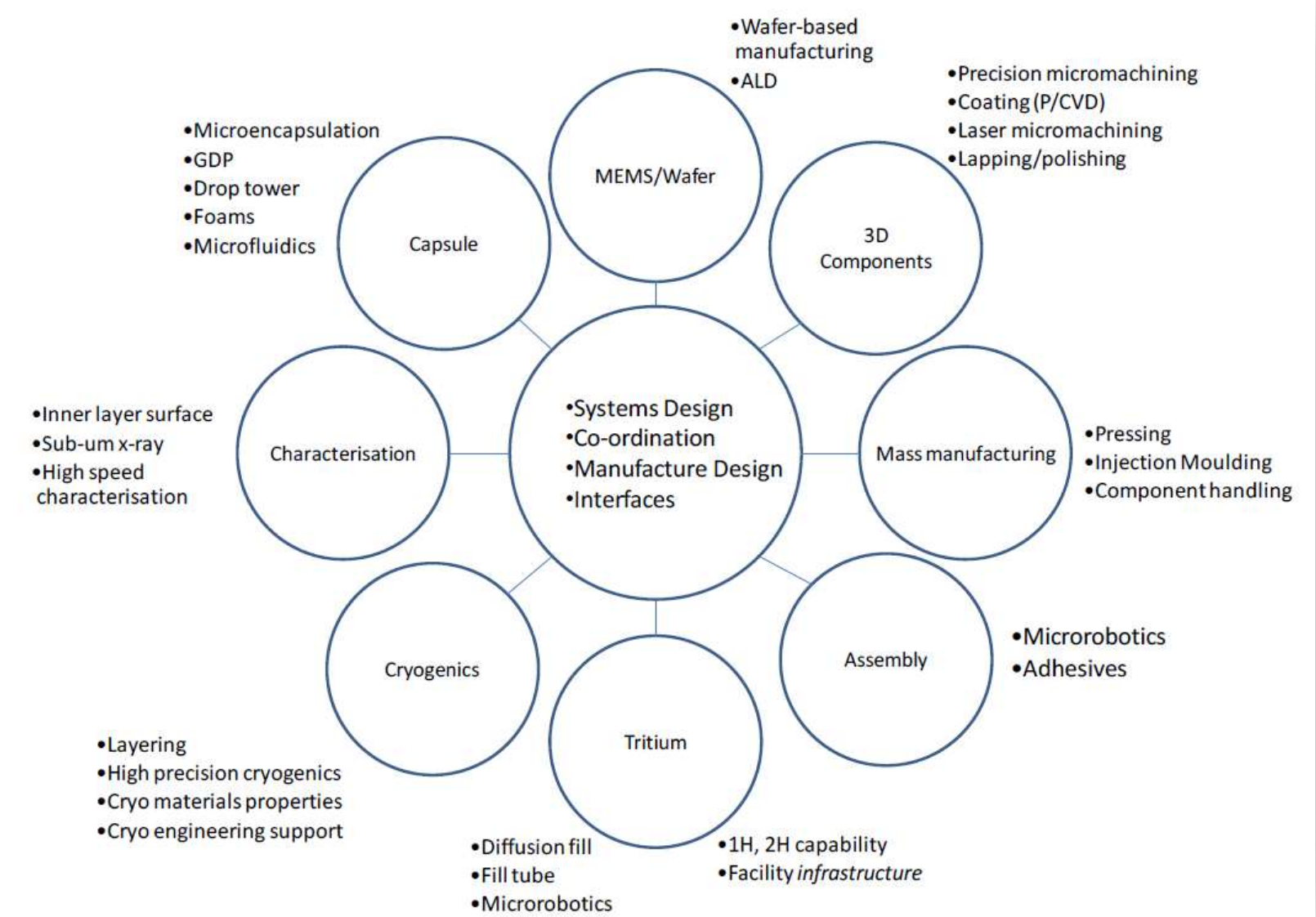
Targetry Business Case Summary



8 key Capability Areas each with sub-capabilites

Summary

- Target technology is one of the key challenges for HiPER

- Considerable progress has been made in the Preparatory Phase in many areas of Targetry

- Building on this work a robust plan for Targetry has been written for the HiPER Business Case

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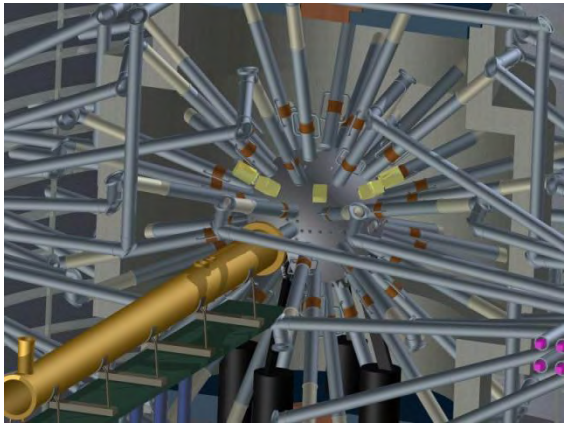
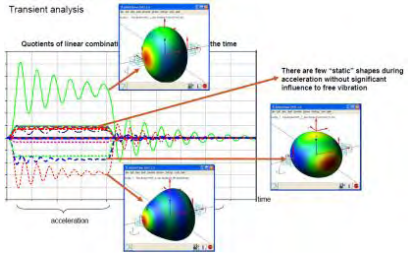
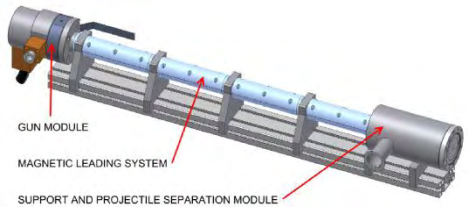
Development of repetition rate fusion target injection for HiPER

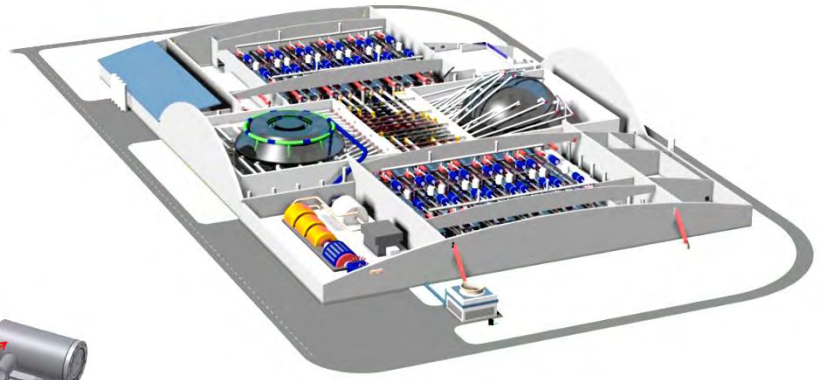
B. Rus[1], P. Homer[1], J. Polan[1], M. Kozlová[1], V. Kolařík[2], T. Papírek[2], P. Havlík[3], M. Kopecký[3], D. Neely[4], M. Tolley[4], N. Alexander[5]

B[1] *Institute of Physics v.v.i. / PALS Centre, 182 21 Prague 8, Czech Republic*
[2] *SWELL s.r.o., Hořice, Czech Republic*
[3] *Delong Instruments s.r.o., Brno, Czech Republic*
[4] *Scientific and Technology Facility Council, United Kingdom*
[11] *General Atomics, USA*









Repetition-rate Target Injection systems

Developed within activities of the Work Package 15 of HiPER including:

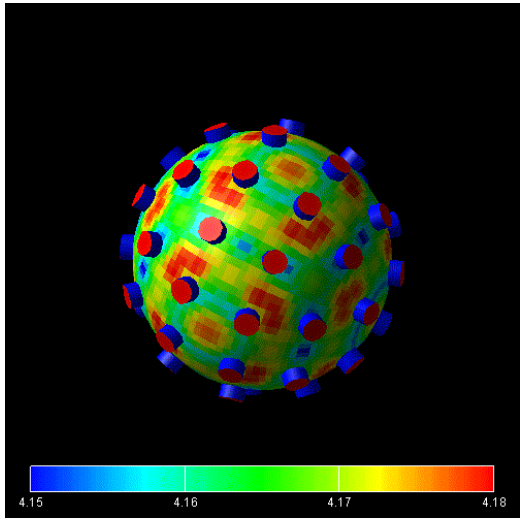
1. Fusion chamber conception
2. Injector schemes and proposed solutions
3. Studies of target integrity during acceleration phase
4. Target chamber and target area shielding
5. Remote handling
6. Debris mitigation, ELMG deflector
7. Time synchronisation between the injected target and laser pulse arrival
8. Baseline target tracking solutions
8. Vacuum system
10. Implosion and fusion diagnostics
11. Maintenance of final optics and diagnostics

Simulations of pellet illumination

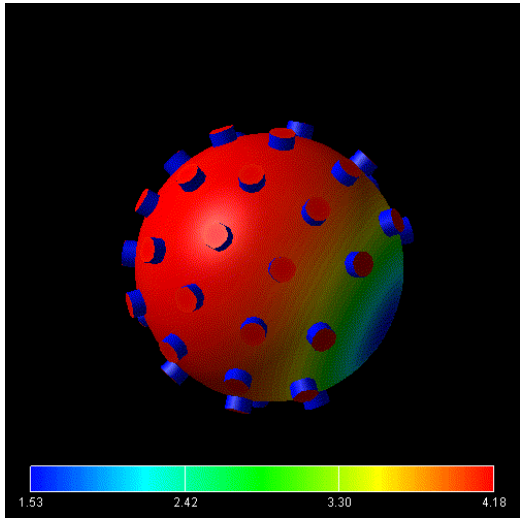
G. Schurtz (CELIA), HiPER Work Package 9

48 beams: perfectly symmetric configuration

Shock ignition



Fast ignition



$$I(\theta) = \frac{1}{\int_{-\pi}^{\pi} r \; d\varphi} \int_{-\pi}^{\pi} I(\theta, \varphi) \; r \; d\varphi$$

This can be expanded in Legendre polynomials :

$$\tilde{I}(\theta) = \bar{I}(1 + \sum_{k=1}^{\infty} a_k P_k(\cos\theta))$$

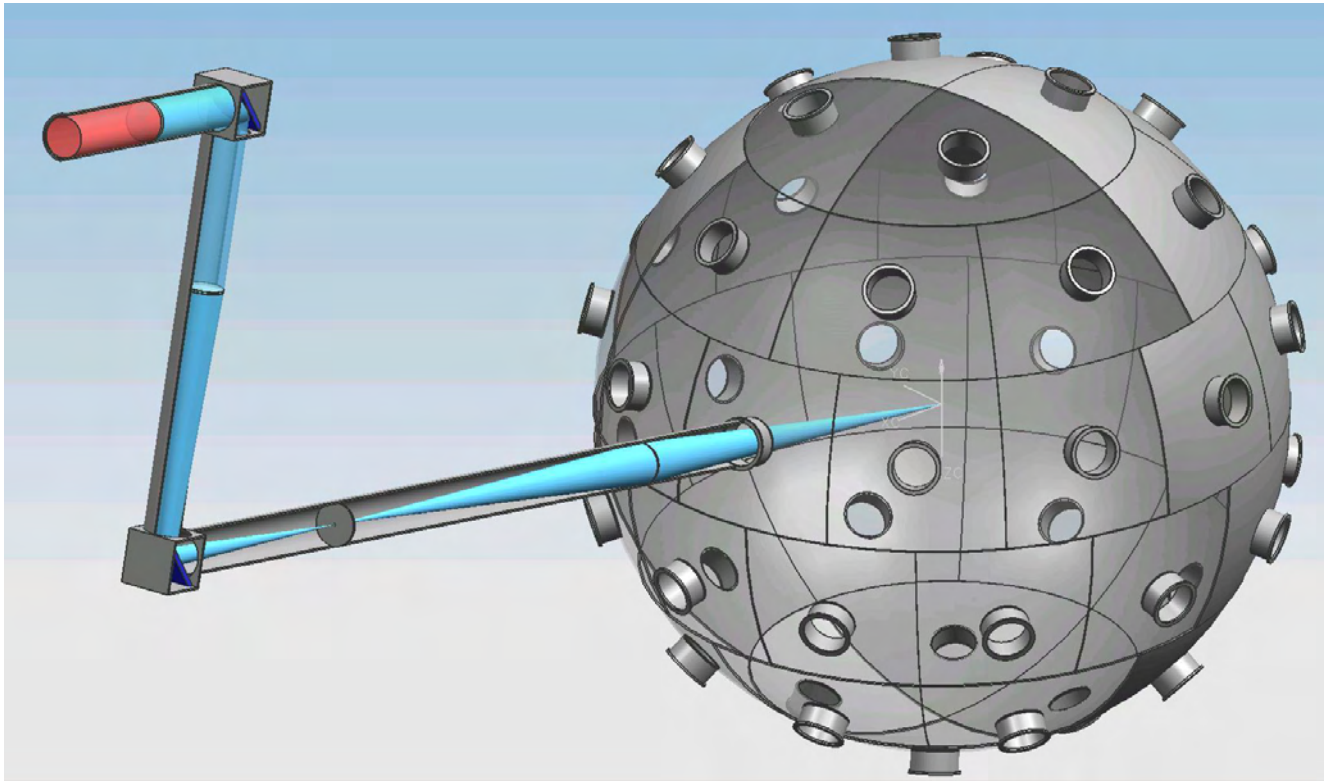
where k is the legendre mode and a_k is calculated using

$$a_k = \frac{2k+1}{2} \int_{-1}^{1} \frac{I(\theta)}{\bar{I}} P_k(\cos(\theta)) \; d(\cos(\theta))$$

a_k is here the normalized legender mode coefficient.

Baseline chamber geometry

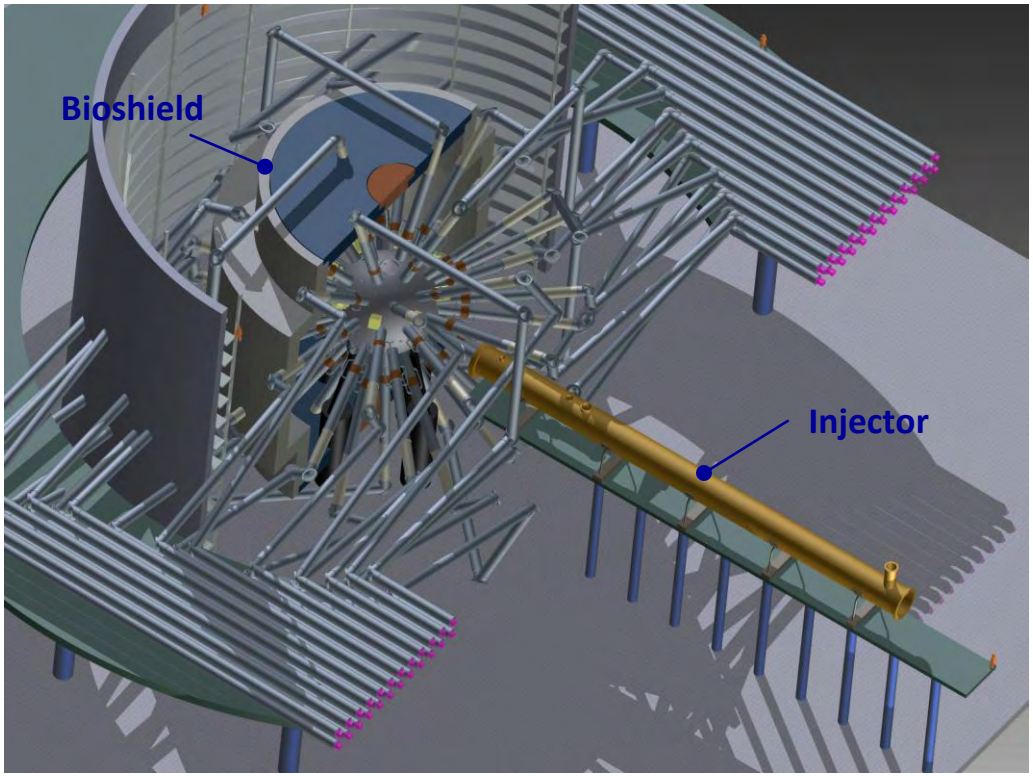
Geometry: 48 beams for spherically symmetric illumination



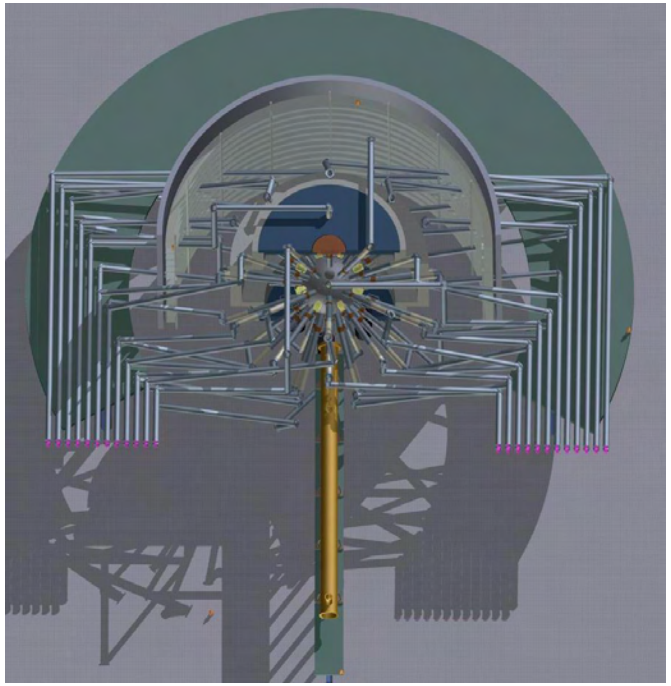
N_b	L	N_i	θ_i (deg)	ϕ_i (deg)	M	σ_{rms}
42	5	5 10 12	25.14 57.33 90.		2	2.46 10^{-2} %
46	4	8 15	30.55 70.12		3	1.5 10^{-4} %
48	12	4	21.23	0.	3	1.77 10^{-5} %
		8(4+4)	47.03	±23.36		
		12(4+4+4)	74.95	0. ±29.83		
60	12	5	21.44	0.	3	2.88 10^{-5} %
		5	41.98	0.		
		10(5+5)	58.84	±23.97		
		10(5+5)	81.27	±12.55		

HiPER chamber geometry design /1

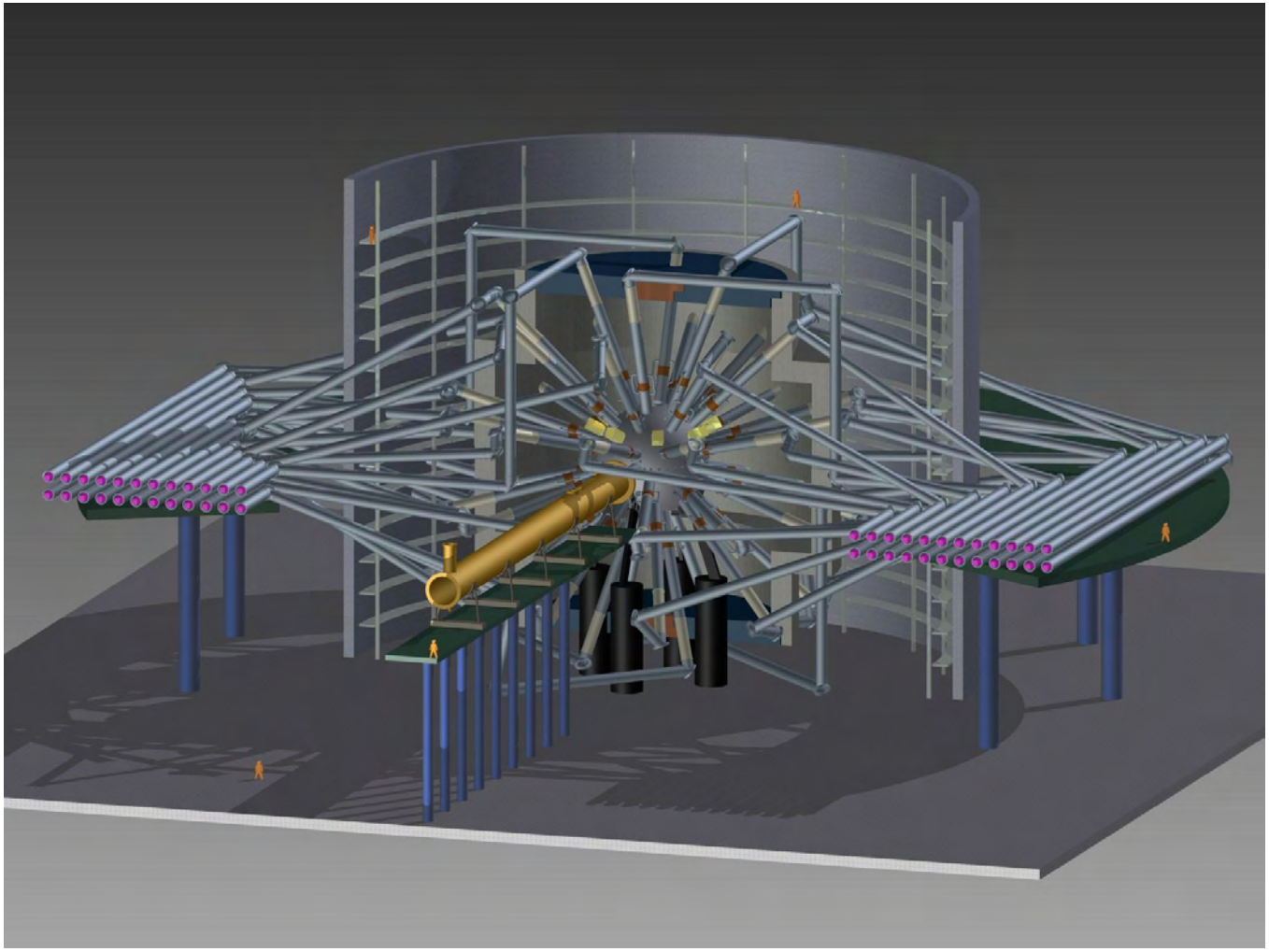
1. Arrival of beams compatible with polar illumination scheme (for HiPER Demo)
2. All mirrors are located outside the bioshield
3. Injector in the horizontal plane, 50 m length, exit plane 10m from the chamber center



Top view

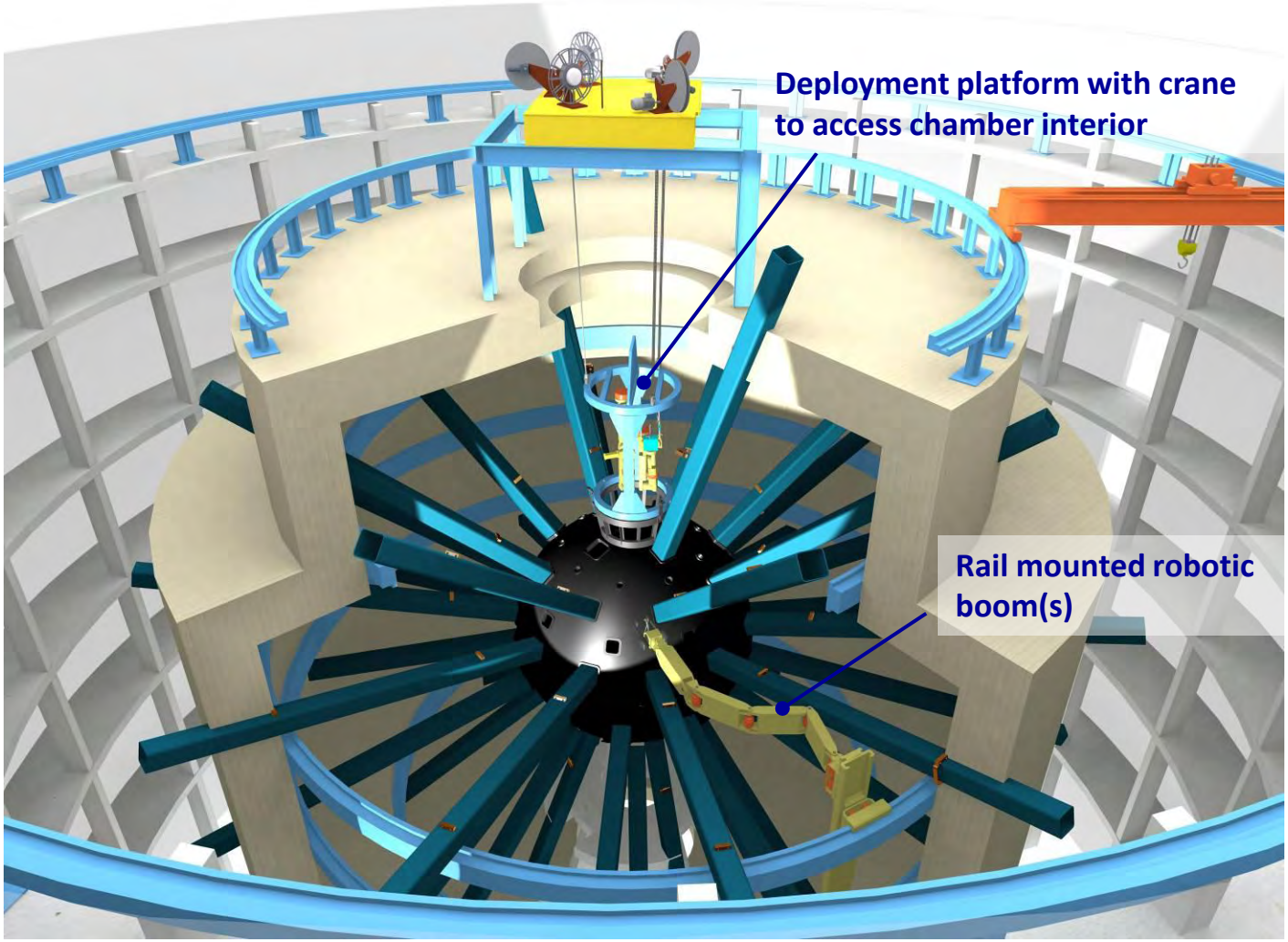


HiPER chamber geometry design /2



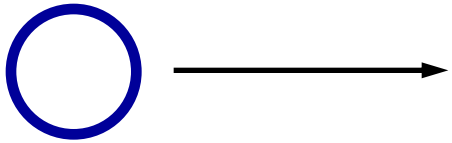
Remote Handling concept for HiPER

Equatorial plane where the injector is placed is unobstructed



Injector design for HiPER

Challenge: acceleration trajectory



v = 500 to 1000 m/s

a < 1000 g

$$s \geq \frac{v^2}{2a}$$

whatever injector design

For v = 1000 m/s: s ≥ 50 m

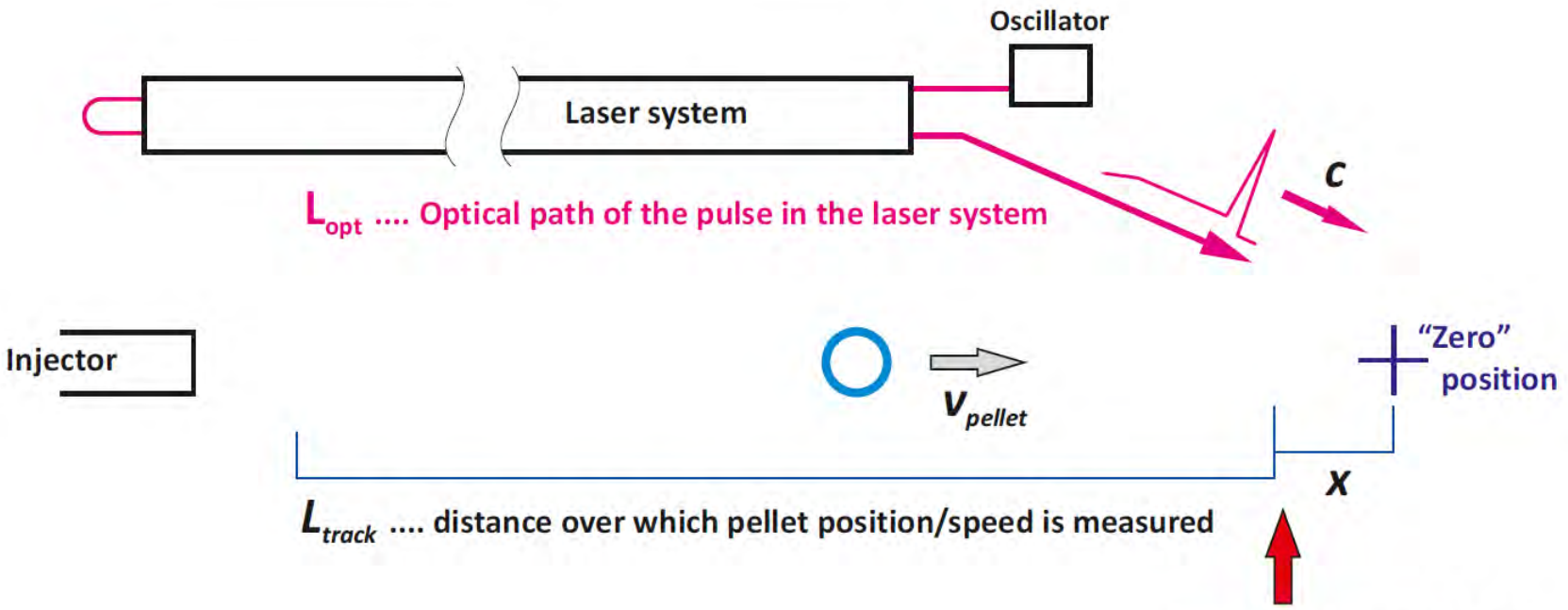
For v = 500 m/s: s ≥ 12.5 m

For v = 300 m/s: s ≥ 4.5 m

Lower injection velocities preferable !

Synchronization of the laser with the injector /1

Laser pulse must meet <u>in time</u> with the injected pellet

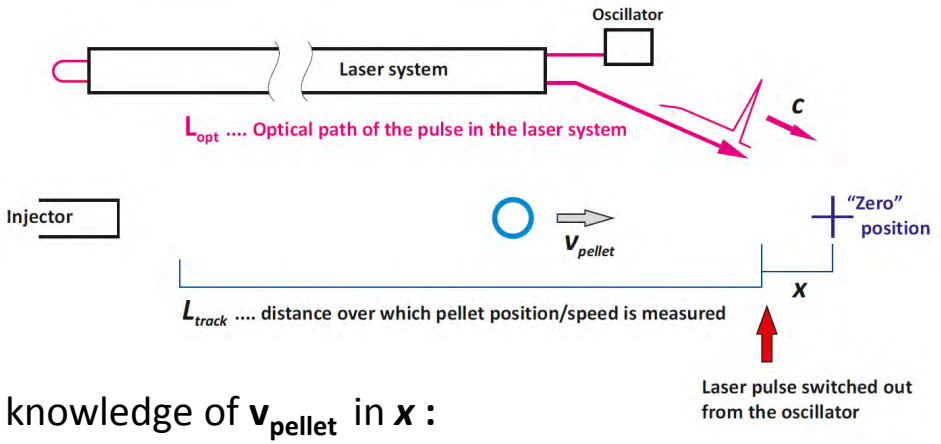


Condition: $\frac{x}{v_{pellet}} = \frac{L_{opt}}{c}$

For $\mathbf{v_{pellet}}$ **= 1000 m/s** and $\mathbf{L_{opt}}$ **= 300 m:** **x= 1 mm**

However, accuracy of the knowledge of $\mathbf{v_{pellet}}$ **in point x is critical!**

Synchronization of the laser with the injector /2



Accuracy of knowledge of $\mathbf{v_{pellet}}$ in $\boldsymbol{x}$:

→ Accuracy of knowledge of its arrival time to "0"

→ Last chance to switch out the laser pulse from the oscillator and to set its arrival to "0"

Target tracking triggers pulse switch-out from the oscillator:

Target arrival prediction accuracy:

$$\Delta t \cong \left(\frac{\Delta v_{pellet}}{v_{0\,pellet}} \right) \frac{x}{v_{0\,pellet}}$$

Sampling/tracking the target trajectory every mm (=1MHz at 1000 m/s) with 10 µm accuracy:

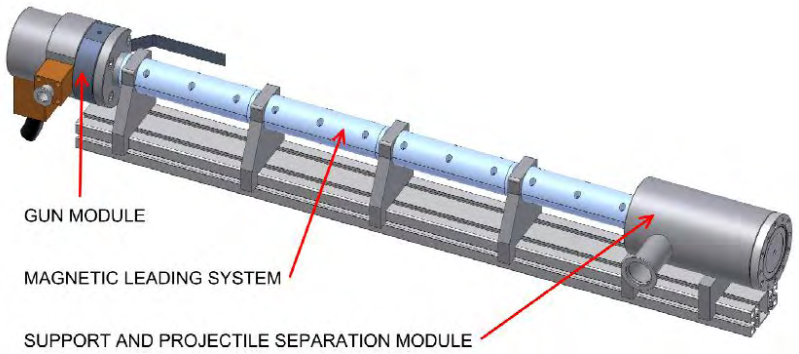
$\Delta v/v = 10^{-2}$: $\Delta t = 10$ ns, $\Delta x = 10$ µm

10 ns uncertainty of target arrival can still be accommodated with pulse-to-pulse distance in the oscillator running at 100 MHz, though better accuracy of tracking would be favourable

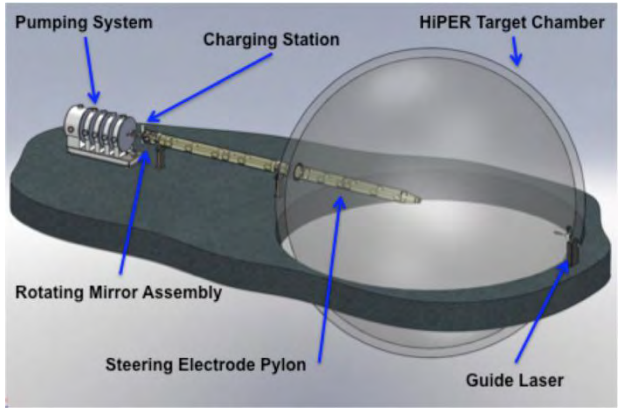
Injector development /1

Search for innovative industry-supplied solutions: 200 kEuro development

1. Injector development & small-scale testbed: Delong Instruments (Czech Rep)



1. Target deflection and steering systems : General Atomics (USA)



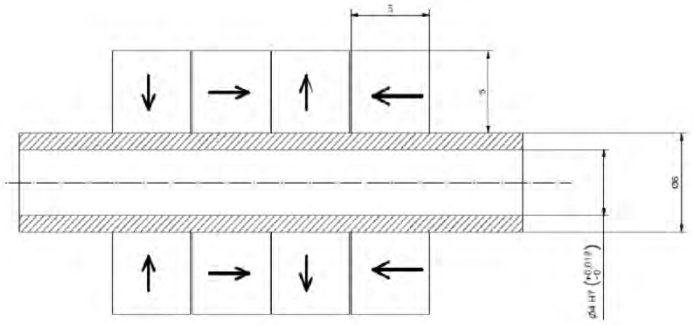
Injector development /2

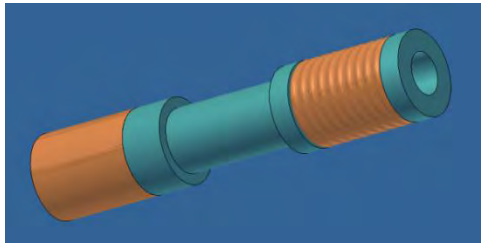
Injector problem separated into two sub-problems

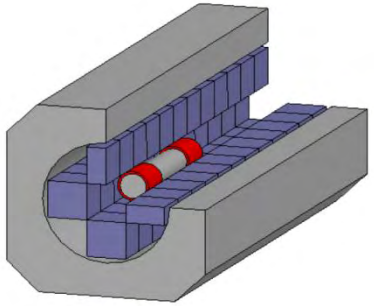
a) Ultra-precise linear guiding + accelerator
b) Sabot-pellet separator, sabot steering

Principle selected for development: MG linear Halbach-type guideway

The guiding is dynamic: induction coils at both end of the sabot



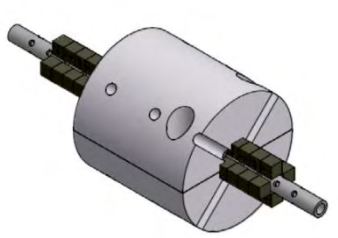




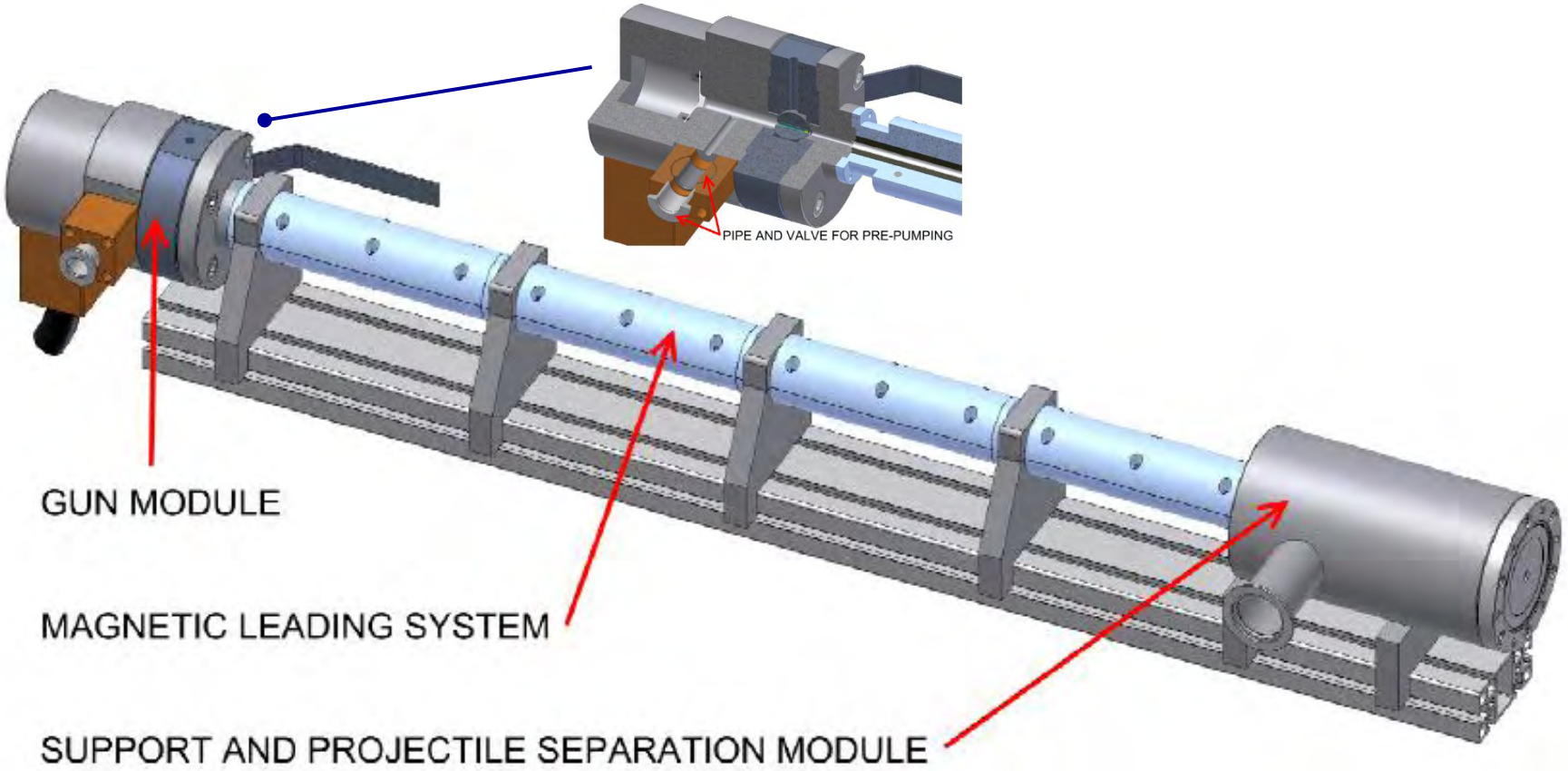
Separated guiding and acceleration

Acceleration at "low speed" (500 m/s) by gas gun

Acceleration at speeds >500 m/s by laser ablation



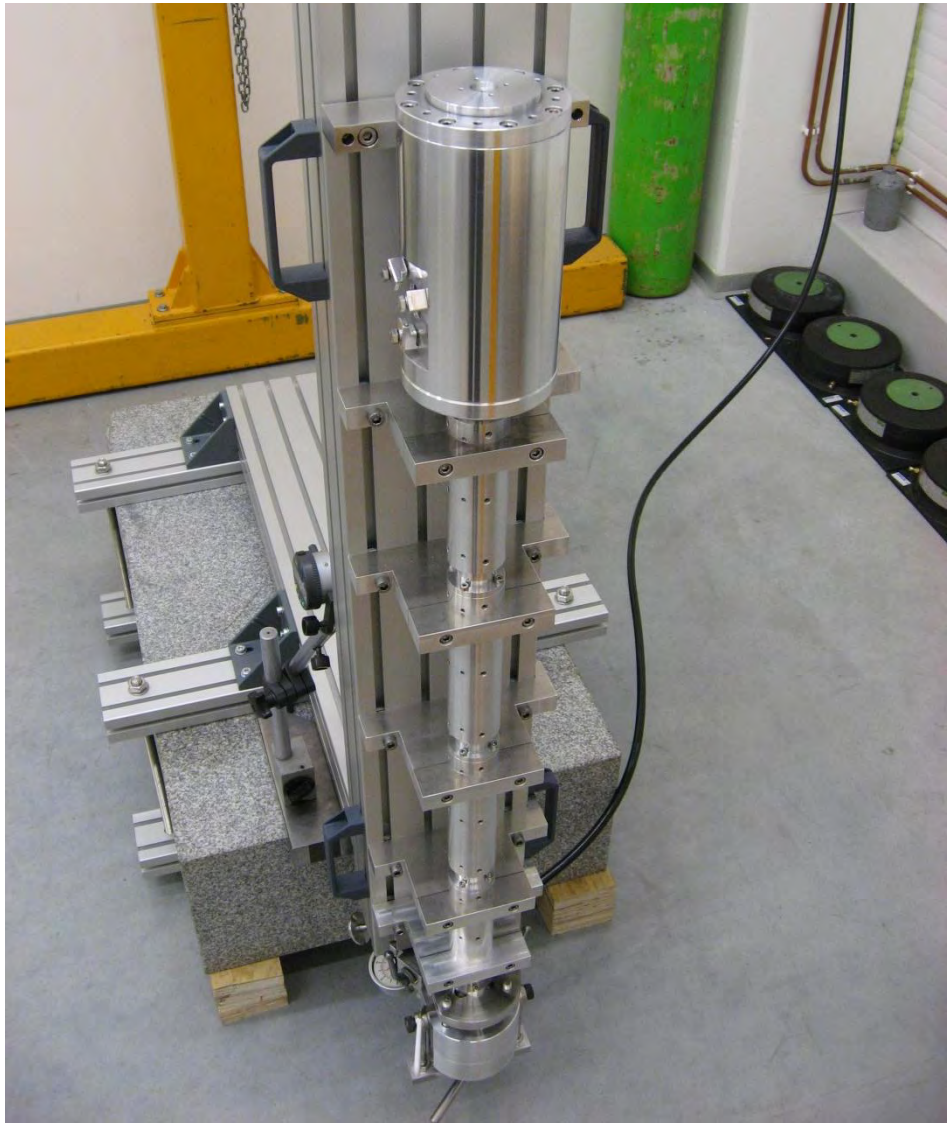
Injector development /2



Construction of a prototype of 1m long module

Gas gun section and precision of a dummy target delivery will be tested

Injector development /3



Injector demonstrator assembling

1 meter long segment of the gas-acceleration section of the injector assembled (January 2011)

Diagnostic package to measure precision of the velocity (high-speed camera) and of lateral guiding (interferometer) being installed (February 2011)

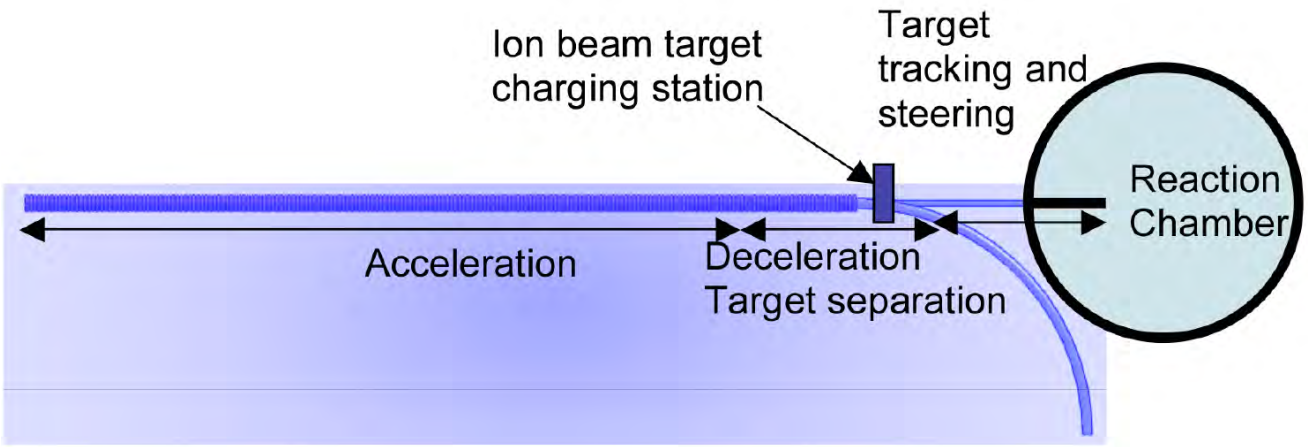
Tests to start in March 2011

Injector development /4

Steering and target-sabot separation:

Four plausible options suggested (General Atomics):

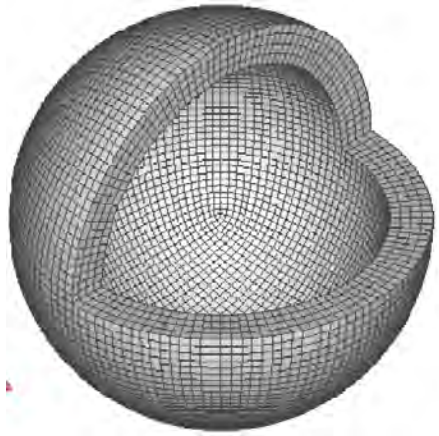
1. **Electrostatic**
2. **Magnetic lens with superconductor layer (Pb) on the target**
3. **Active magnetic steering**
4. **Laser ablation**



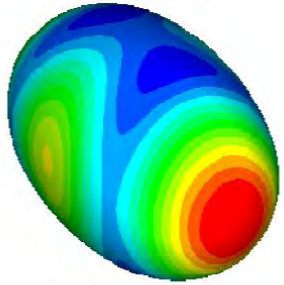
Studies of target deformations during injection

Study of target shape during acceleration: finite element analysis
2-mm diameter pellet studied (shell+cryogenic D-T)
0.2mm DT fuel layer thickness, 0.74mg mass

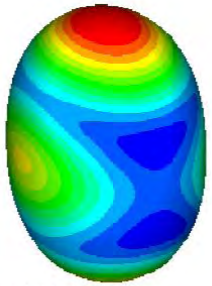
Pellet FEM



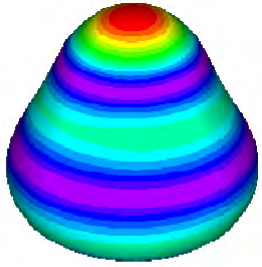
Lowest modes of oscillations:



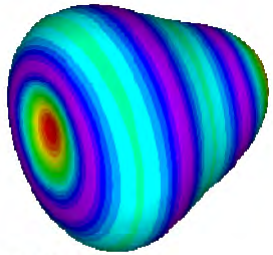
Output Set: Mode 1, 1352.496 Hz
Deformed[1.081]: Total Translation
Contour: Total Translation



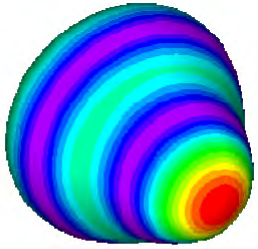
Output Set: Mode 2, 1352.504 Hz
Deformed[1.083]: Total Translation
Contour: Total Translation



Output Set: Mode 6, 1827.544 Hz
Deformed[1.083]: Total Translation
Contour: Total Translation



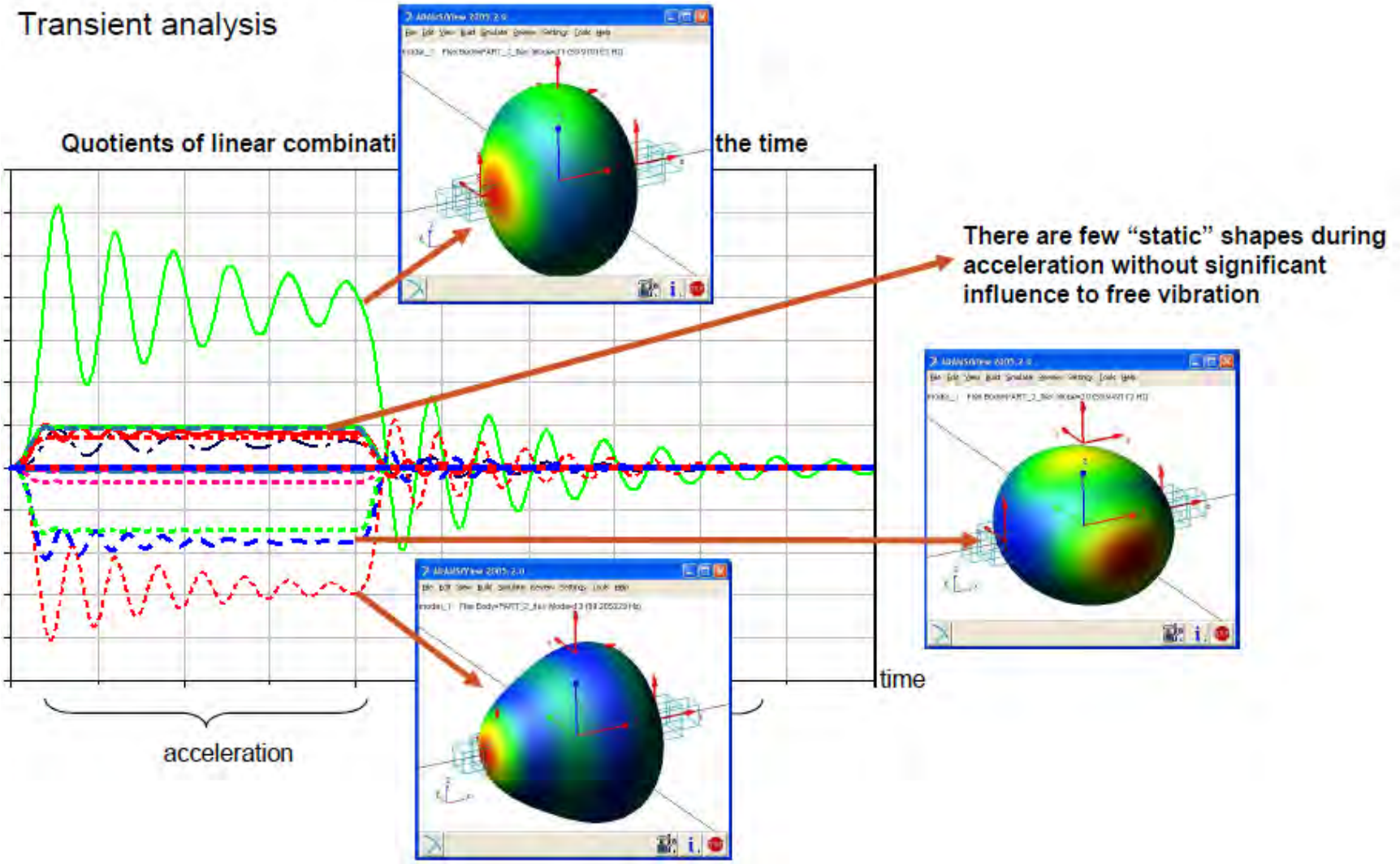
Output Set: Mode 7, 1827.555 Hz
Deformed[1.051]: Total Translation
Contour: Total Translation



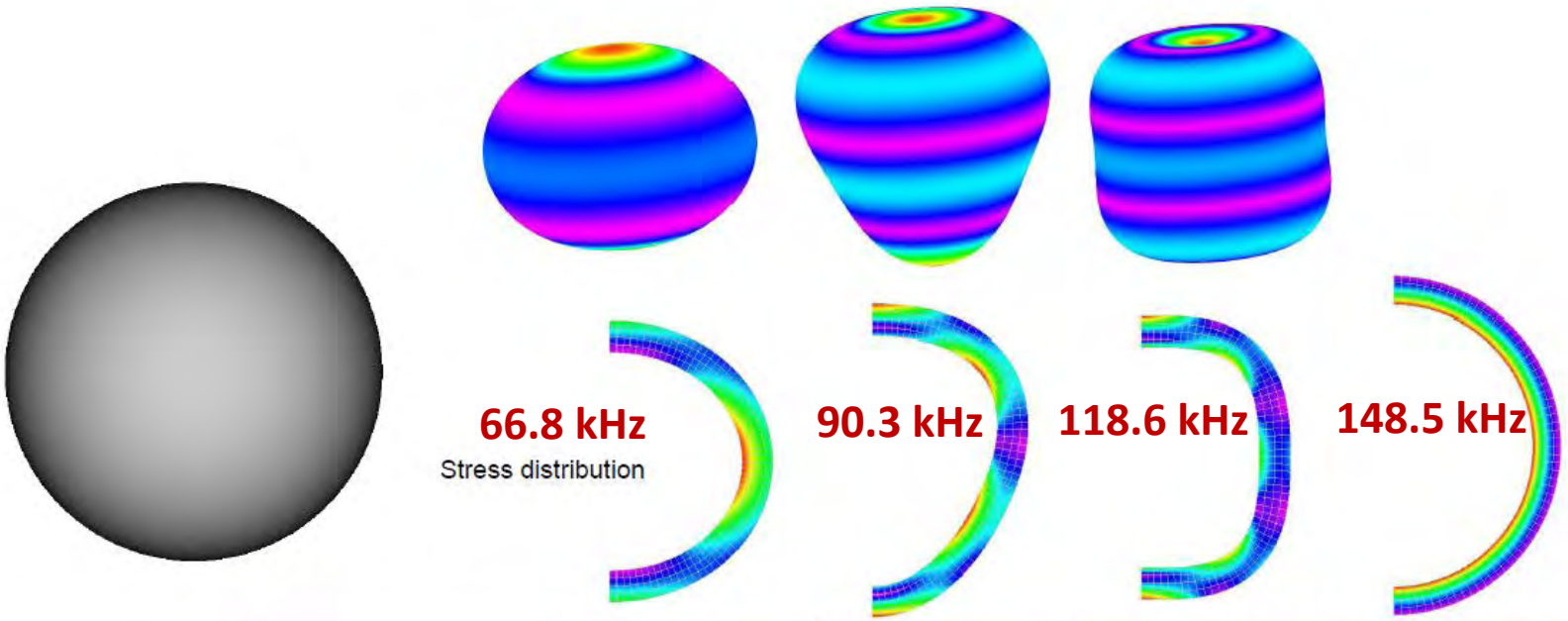
Output Set: Mode 8, 1827.585 Hz
Deformed[1.391]: Total Translation
Contour: Total Translation

Oscillation modes excited during injection

Acceleration 1000 g over 50 m to the speed 1 km/s



Pellet deformations



	Eigen shape No.1 „cigar-lentil“	Eigen shape No.2 „pear“	Eigen shape No.3 „cylinder“	Eigen shape No.4 „breathing“
Frequency	66858 Hz	90329 Hz	118629 Hz	148512 Hz
At maximal stress σ_{HMH} = 90 kPa will be maximal displacement	u = 1.17 µm	u = 0.98 µm	u = 0.67µm	u = 0.57 µm

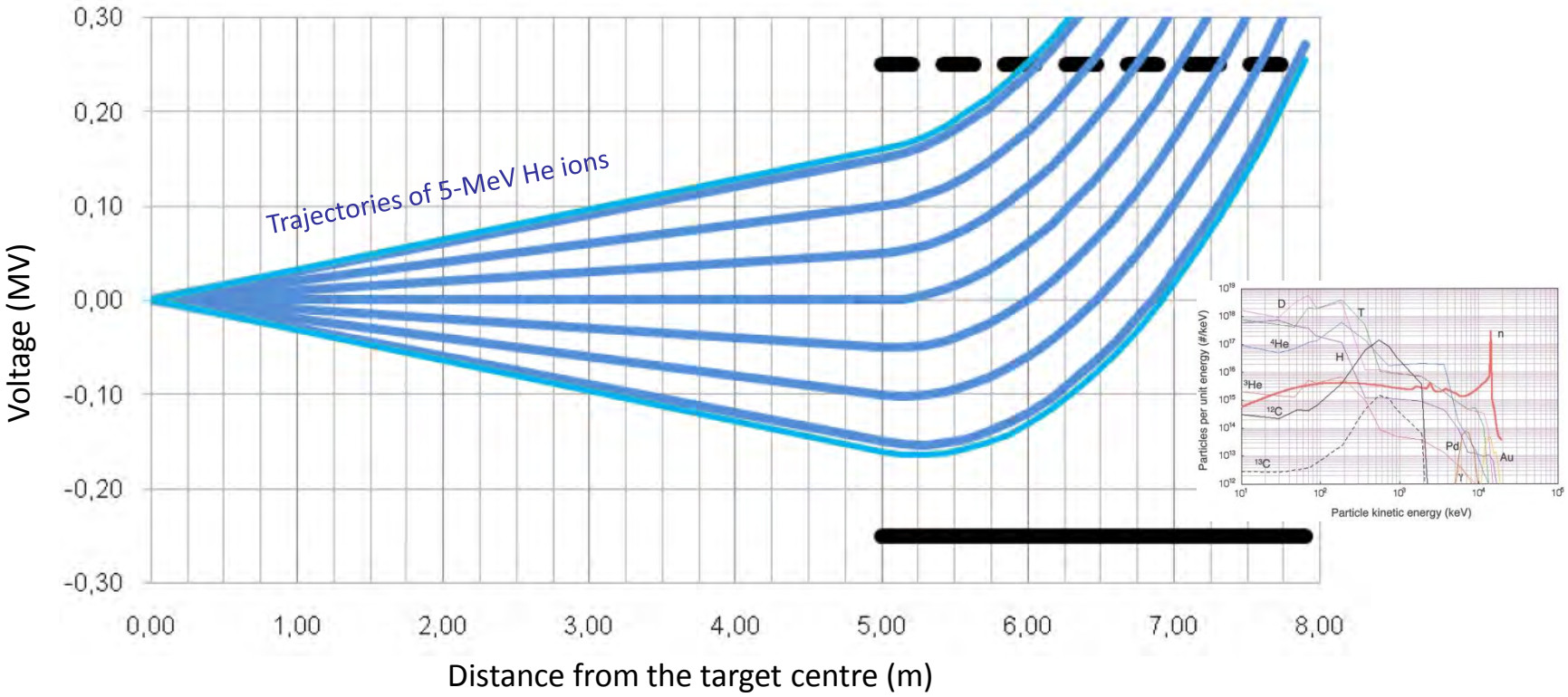
Deformations of the pellet accelerated at 1000g upon its arrival to the chamber center are <1 µm

Simulations of FI and indirect-drive targets underway

Fusion debris deflector study

Electrostatic deflector: preferred over magnetic solution due to field magnitude

2.9 m long electrodes located between the final lens and the chamber perimeter



Potential of collection of unburned T on the negative electrode!

Conclusions

Potential solutions addressing critical issues of repetition rate target injection identified

There seems to be no conceptional show-stopper for target injection at repetition rate of 10 Hz

Prototyping of the injector, of the steering systems and achieving target tracking accuracy 10 microns or better critical for demonstrating viability of the suggested solutions

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N.G. Borisenko[1], A.I. Gromov[1], Yu.A. Merkuliev[1], A.S. Orekhov[1]
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METALS PRODUCED AS NANO-SNOW LAYERS AND AS NANOPARTICLES SUSPENDED IN POLYMER FOAM FOR CONVERTERS OF LASER LIGHT INTO X-RAY FOR INDIRECT TARGETS AND FOR INTENSIVE EUV SOURCES.

MSU
Faculty of Physics

P. N. Lebedev
Physical Institute

OUTLINE

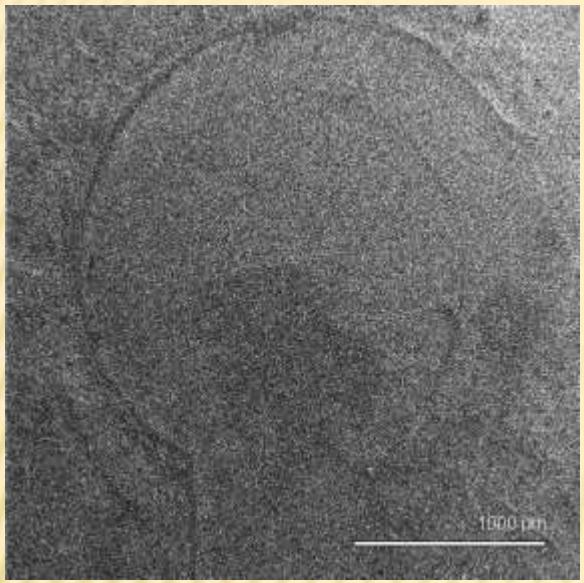
- Low-density nano-snow metal layers down to 1% of solid material density are considered. Specification of such structures and their parameters is required for pre-experimental non-destructive target characterization. The intrinsic structures have nano-sized average cells.
- Such low-density structures can be used inside indirect laser targets and as a cover of inner walls of advanced hohlraum to increase radiation temperature and x-ray emission.
- Searching for better x-ray emitters in plasma polymer aerogels with metal nanoparticles are discussed.

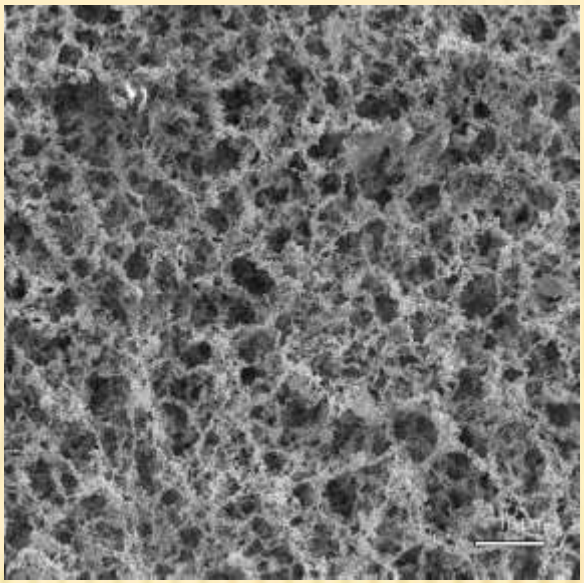
NANO-SNOW LAYERS FABRICATION

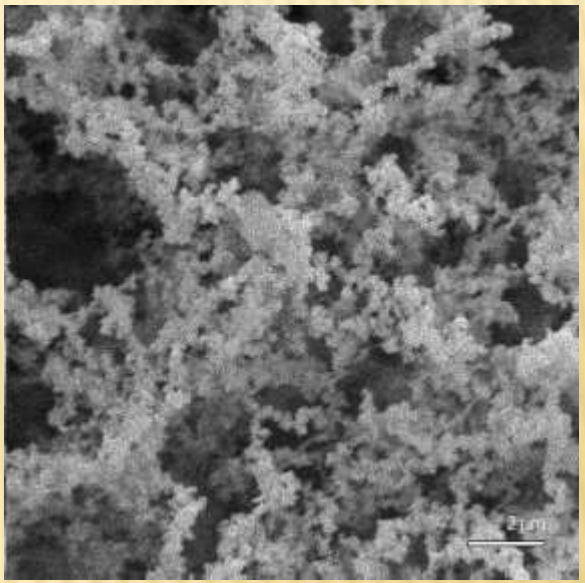
Dr. Gromov with the metal foam production facility (upper)

- Set-up scheme:
- 1 –vacuum vessel,
- 2 – bottom,
- 3 –vacuum and gas pumps,
- 4 – vessel with heated metal,
- 5 – heat shield,
- 6 – targets, 7 – “witness”.

BISMUTH TARGETS

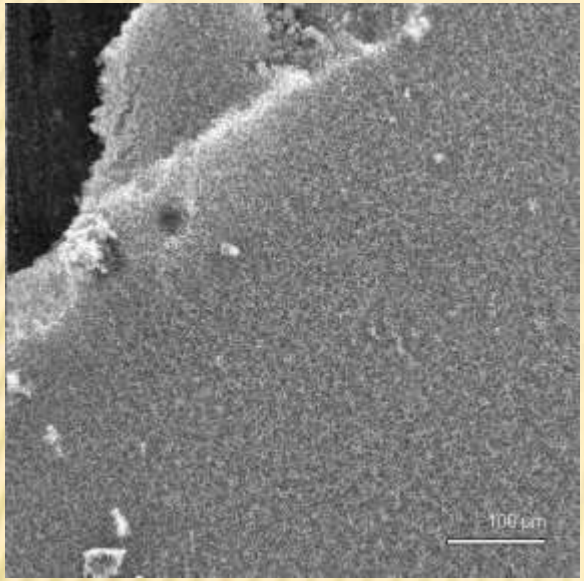


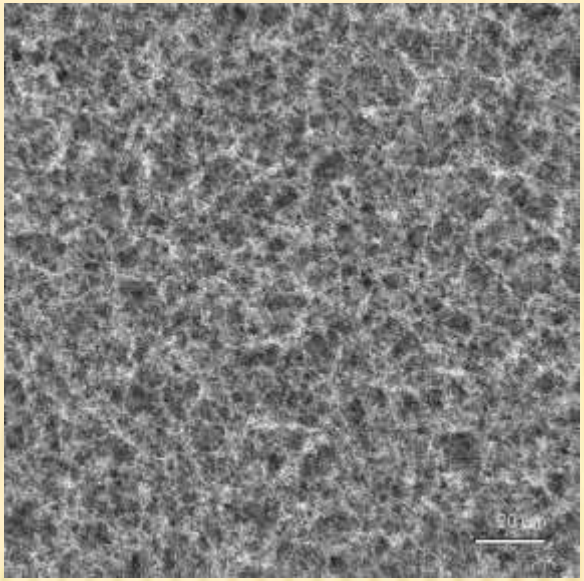


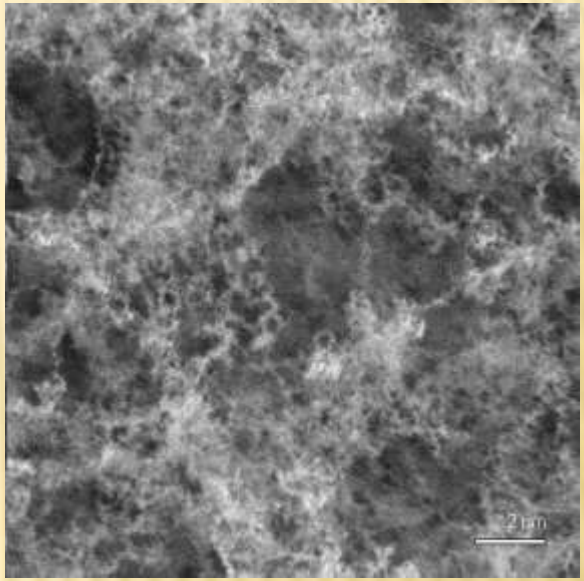


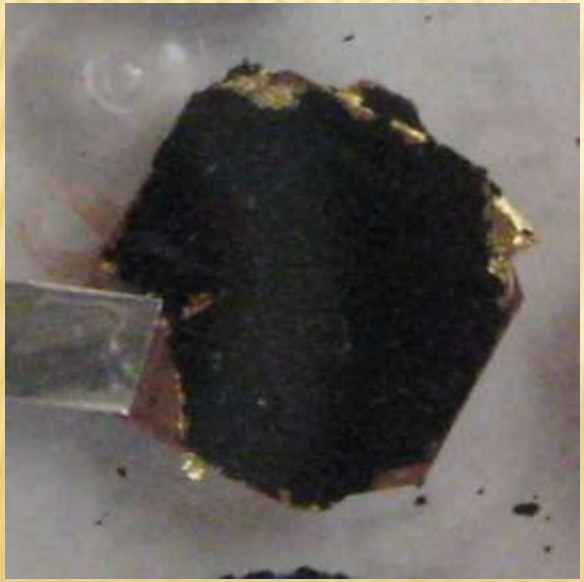
- SEM images of the Bi target. Homogeneity and similarity of the sample structure is visible on different scales. (upper)
- Bi targets 80 and 135 mkm thick. (left)

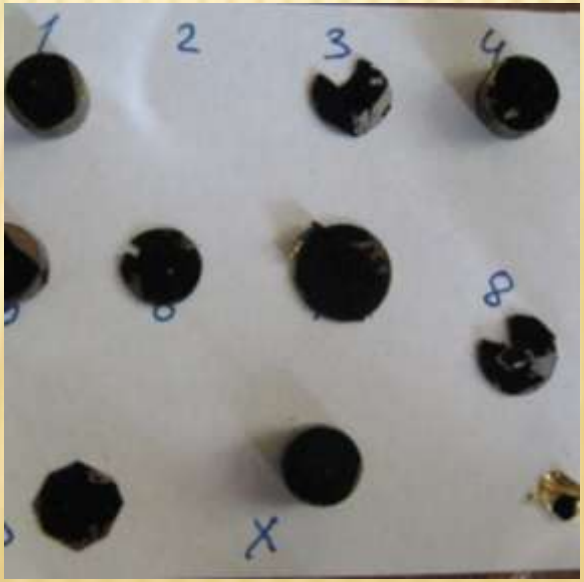
GOLD TARGETS





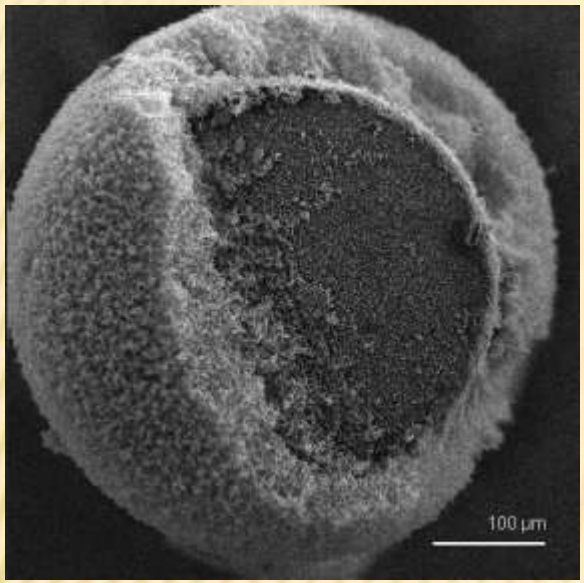


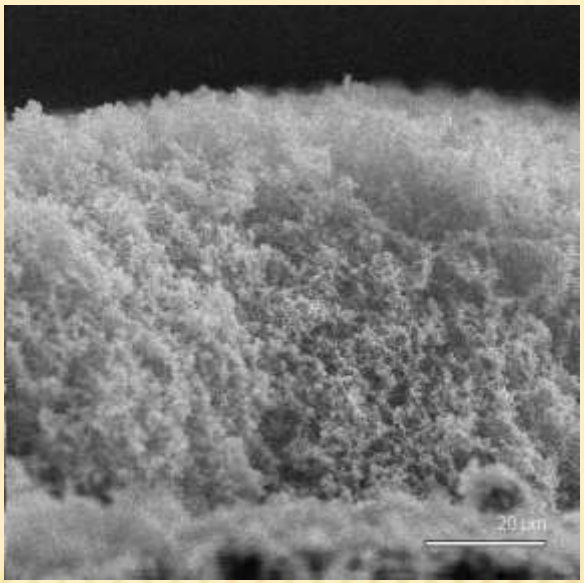


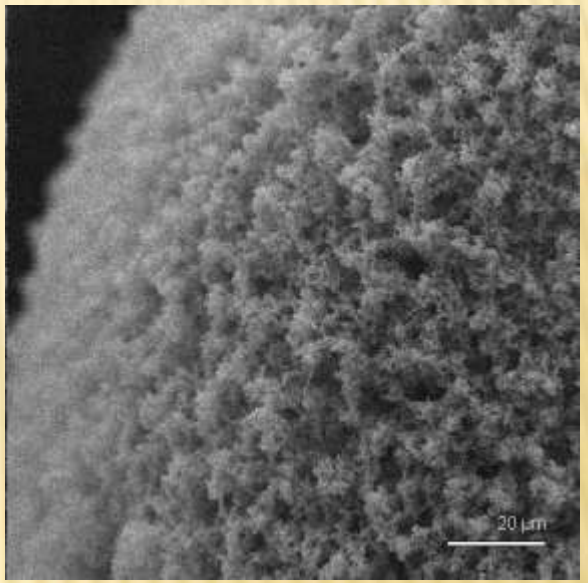


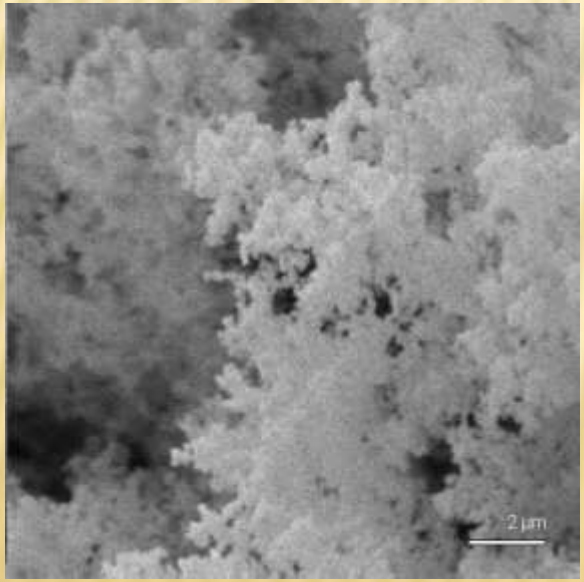
- SEM images of the Au target. The intrinsic structure on different scales resembles Bi one which is due to similar Z number. (upper)
- Au targets of various properties. (left)

SEM IMAGES OF THE "WITNESS"



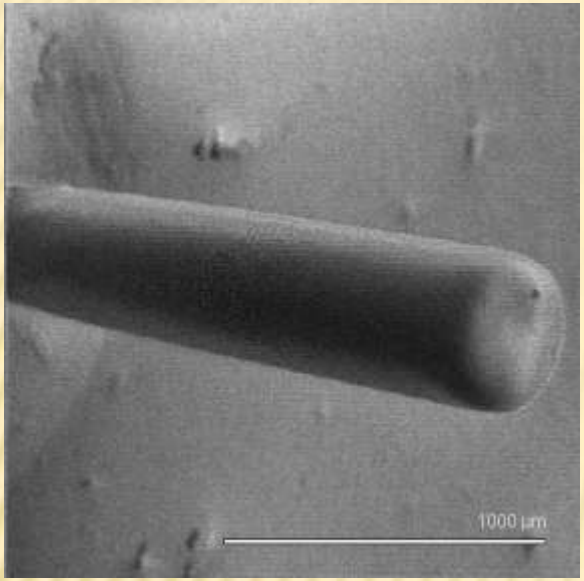


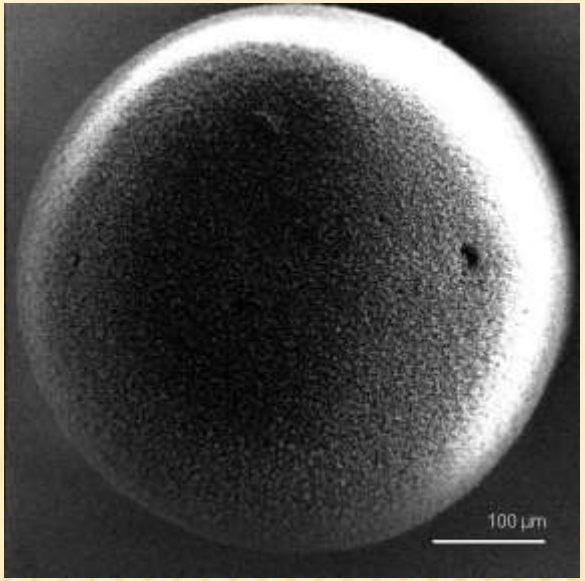


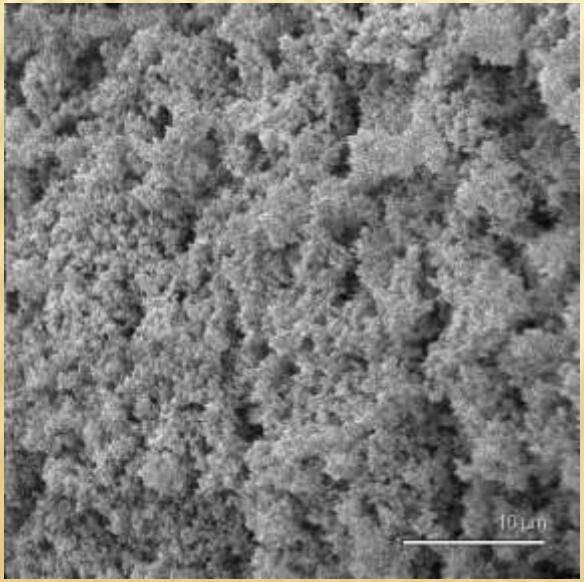


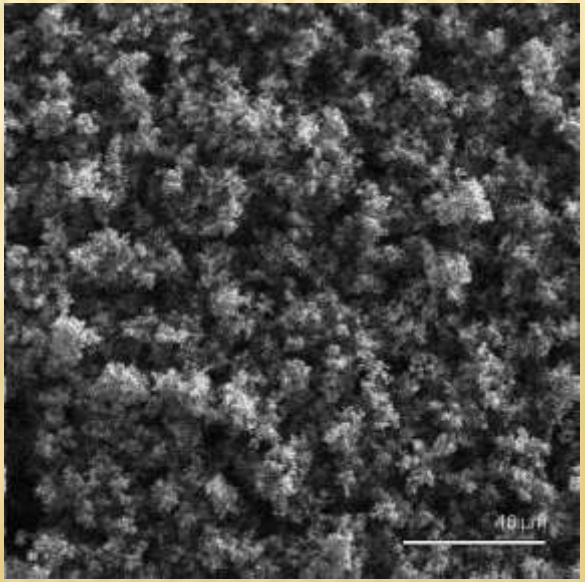
- Velocity *V* of nanoparticle precipitation in the low-pressure camera can be estimated with the Stokes formula as viscous movement. If considering nanoparticles as spheres and the drag force as opposite to gravity force on a sphere (r – radius, ρ – density),
- $V = 9\rho g r^2 / 2\eta$
- where g – free fall acceleration, η – viscosity coefficient.
- Velocity of 100 nm solid bismuth nanoparticle in ~500 Pa Ar atmosphere is approximately 2 cmph. So collection of all the particles of the metal "fog" down to 10 nm in diameter would take more than a month.
- Due to wide distribution of nanoparticles by the weight their interaction during precipitation leads to formation of fibers of nanoparticles and claws of fibers. However the distance between nanoparticles (camera 0.05 m^3, 5 g of metal =>17 mkm) is considered unpleasant for any significant aerodynamical forces during similar spheres' fall.
- The structure of nano-snow layer on the graphite cylinder is the same as on the target.

SEM IMAGES OF THE "WITNESS"







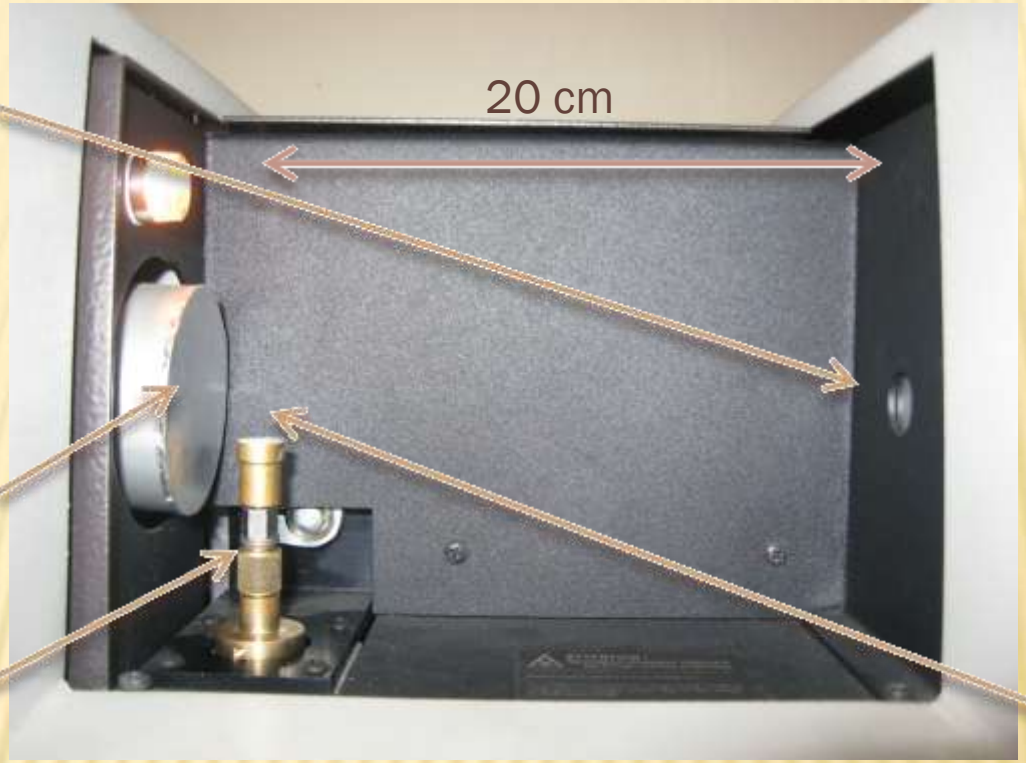


- Diameter of nanoparticles in the nano-snow layer and the layer density depend on the gas pressure in the camera and the gas itself.
- Low-density Bi layers are rather fragile and can sinter at the temperatures around 50-70 C.

SKYSCAN 1074 MICROTOMOGRAPH

X-ray tube window
- U=20-40 kV
- A_{max}=1000 μA
- Ø = 100 μm

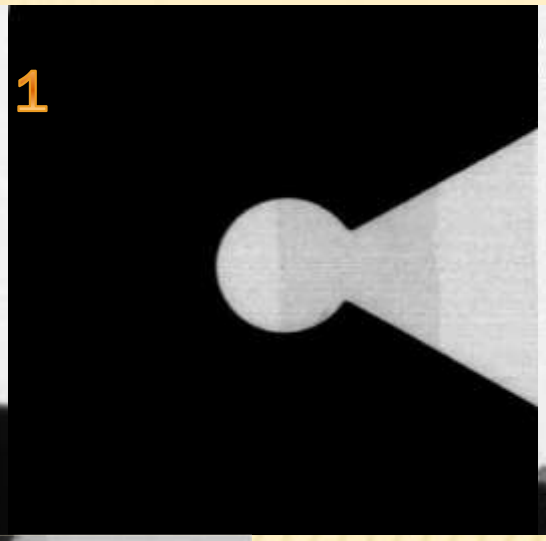
CCD-camera looking at phosphor screen under thin Al filter
- Pixel size 21 μm
- 12 bit depth

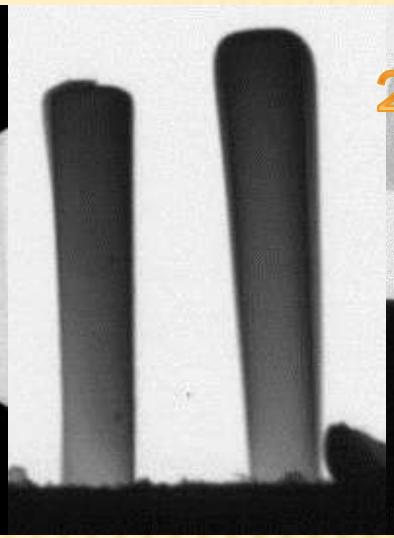


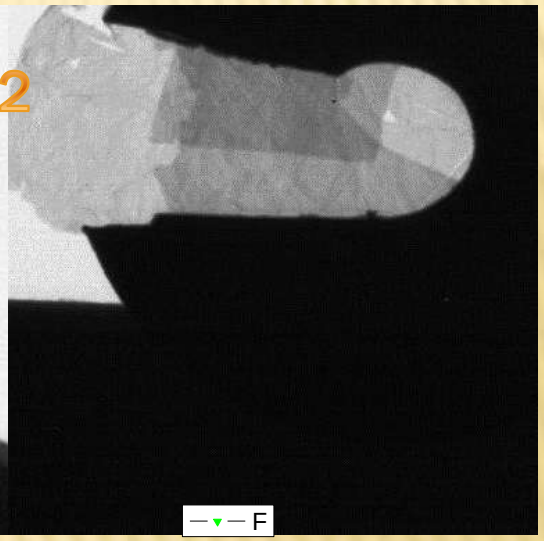
Au “witness” on the holder.

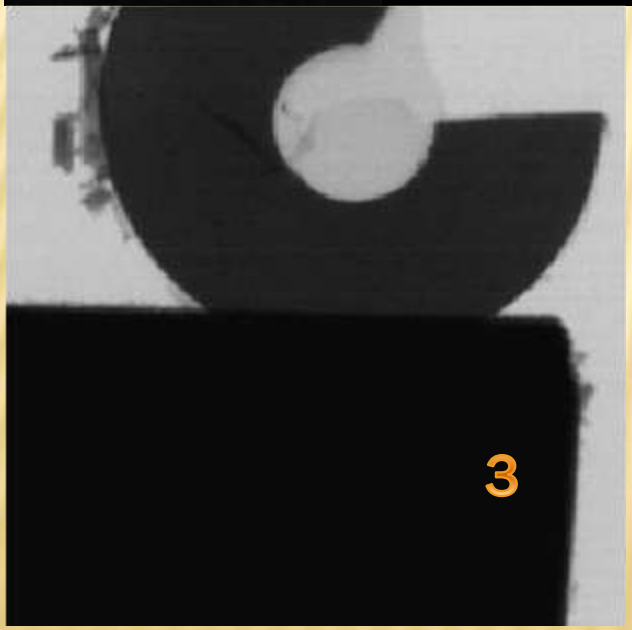
Rotating table for the samples. Minimum rotating angle – 0.9° (400 steps per 1 full turn)

TARGET CHARACTERIZATION



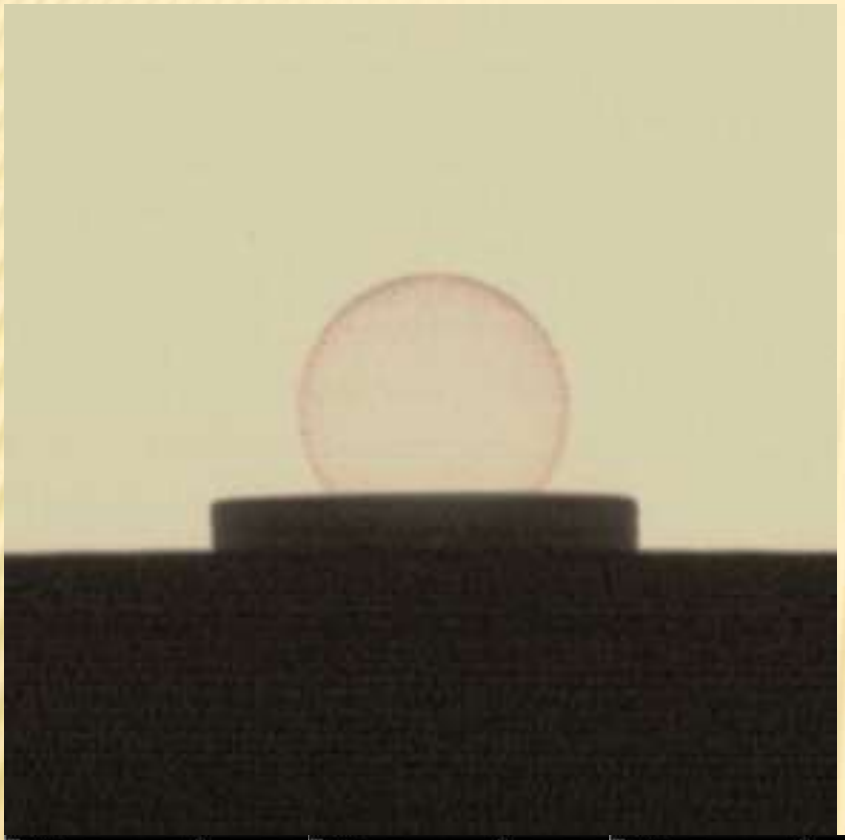




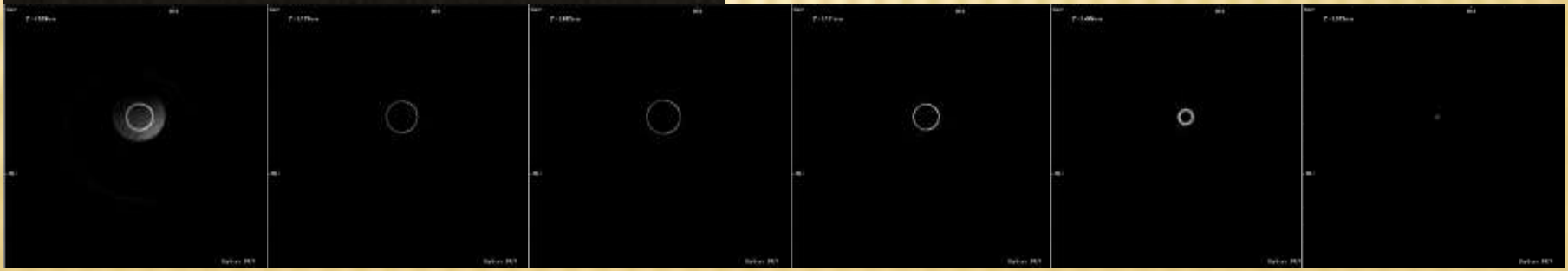


Material	Thickness	Absorption ratio, cm^{-1}
Bismuth	0.25	1428
	0.50	1418
	1.0	1442
	2.0	1452
	3.0	1453
Gold	0.14	3094
	0.28	3176

PRECISION INCREASE



- Additional information about the target can be used for precision increase. However, an ill-defined problem is solved only for a flat picture. With given devices additional manipulations introduce errors of the same or even lower order of accuracy as the achieved refinement if solving the problem for 3D.



TARGETS WERE USED IN LASER-PLASMA EXPERIMENTS

- Experimental study of conversion of laser radiation (λ=1.05 mkm, 10^{14} W/cm^2, pulse duration 0.5 ns, energy 15 J) into x-rays in solid and in low-density bismuth indicated 10% increase of conversion ratio in nano-snow layers in comparison with solid metal layers. The spectrum becomes softer. [ref]

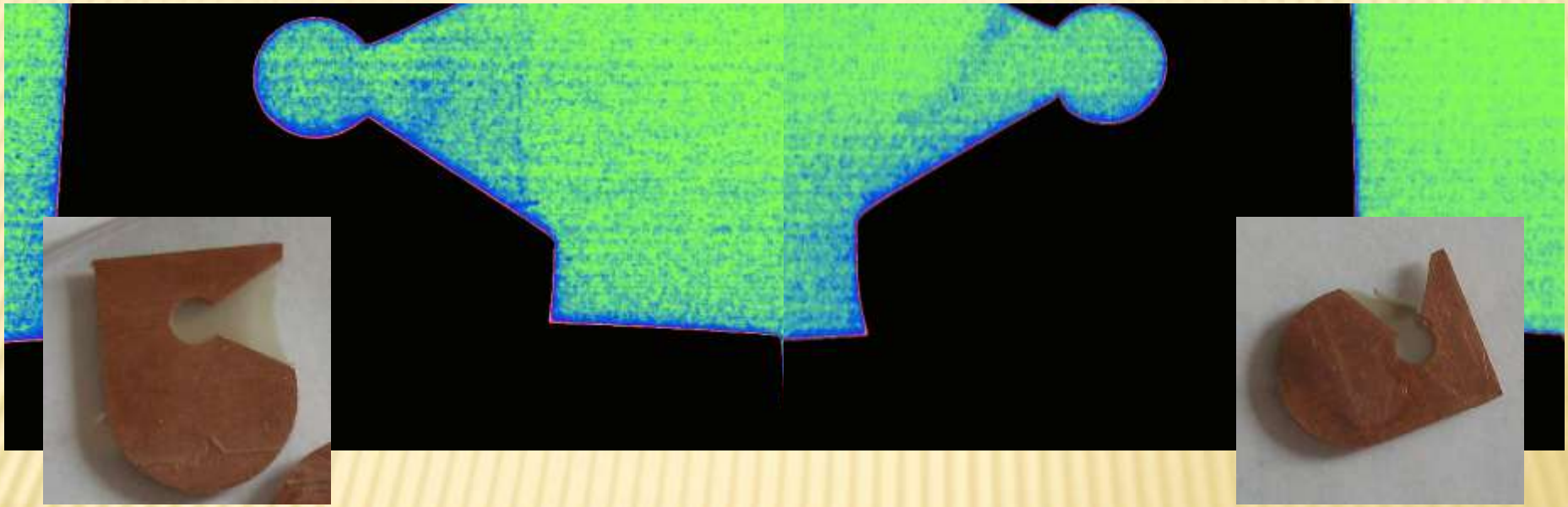
[ref] N.G. Borisenko, A.I. Gromov, Yu.A. Merkuliev, A.S. Orekhov, S. Chaurasia, S. Tripathi, D.S. Munda, N.K. Gupta, L. Dhareshwar. Comparison of laser light conversion efficiency into x-rays in solid bismuth and in low-density bismuth.// Preprint of the P.N. Lebedev Phyisics Institute, 2011, N29.

TAC AEROGEL FABRICATION

1. Solution
2. ! Ultrasonic agitation with M-nanoparticles
3. 2 Gelation initiated
4. 3 Solvent exchange
5. 4 Critical point drying
6. Polymer aerogel in the washer [ref]

[ref] Borisenko N.G., Khalenkov A.M., Kmetik V., Limpouch I., Merkuliev Yu.A., Pimenov V.G. (2007). Plastic aerogel targets and optical transparency of undercritical microheterogeneous plasma.// FuSci and Tech. 51, 655-664

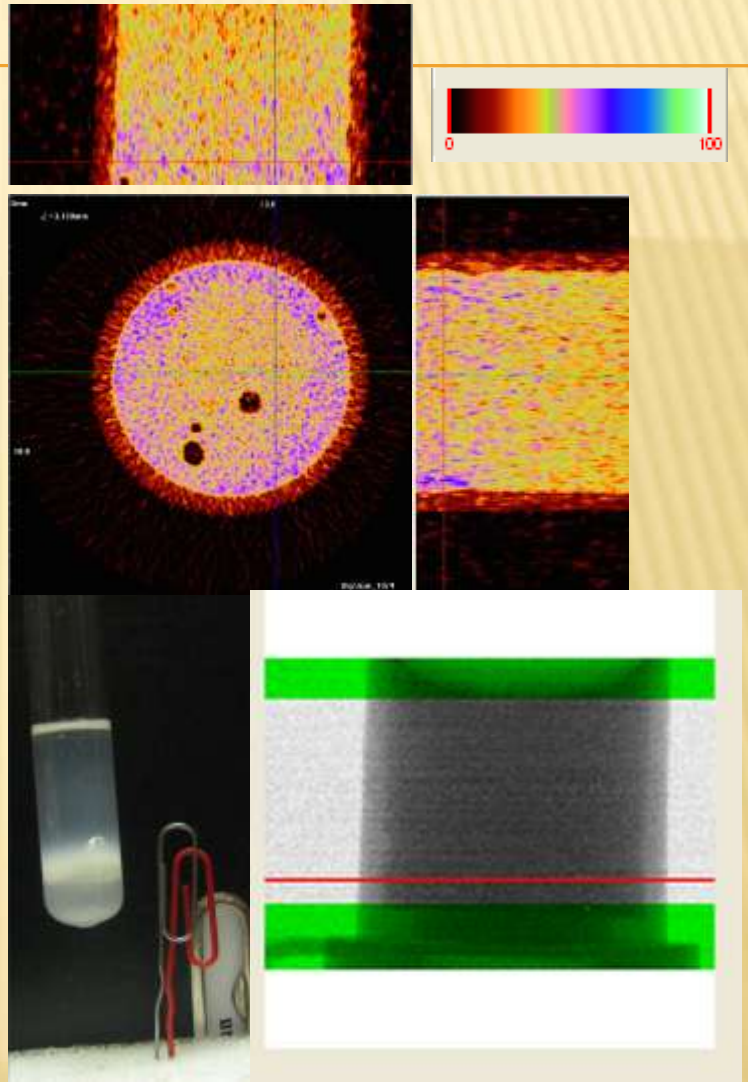
CU-DOPED (10%) TAC AEROGEL



500 mkm thick Cu-doped (10%) TAC foams, 6 mg/cc, are visible on the microradiogram. Defect of density non-homogeneity on the edge of the foam is visible on the right picture. Method of tomograph calibration and final target characterization is to be developed.

CONCLUSION

- An additional method of target characterization has been added to earlier existing ones in Thermonuclear Target Laboratory, LPI, and is now being developed. The data on x-ray optical density of the material is provided by the microtomograph SkyScan 1074 then calibration and mathematical processing are used to achieve such target characteristics as density and layer thickness.
- For nearly transparent targets defects of structure are visible. Optical density and density gradient can be observed on the scale of ~20 mkm. With mathematical methods boundaries may be characterized more precise than 1 pixel value (precision ~6 mkm).
- Earlier the commercial tomograph was proposed and used for samples of the scale of 1 cm. At present submillimeter foam and aerogel samples are characterized successfully.(See also the presentation of A.Orekhov et al on this workshop)

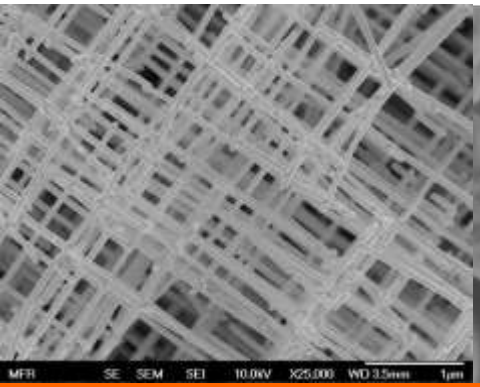


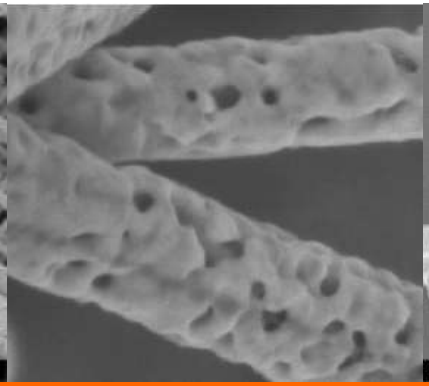
THANK YOU FOR YOUR ATTENTION

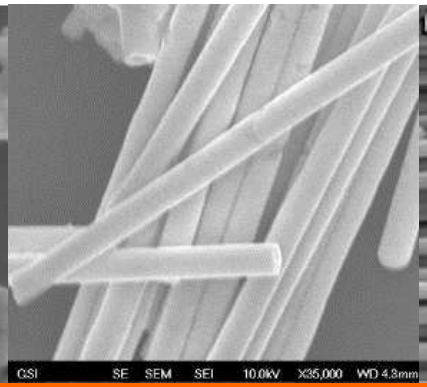
Micro- and Nanowire Arrays by Heavy-Ion Irradiation and Electrodeposition

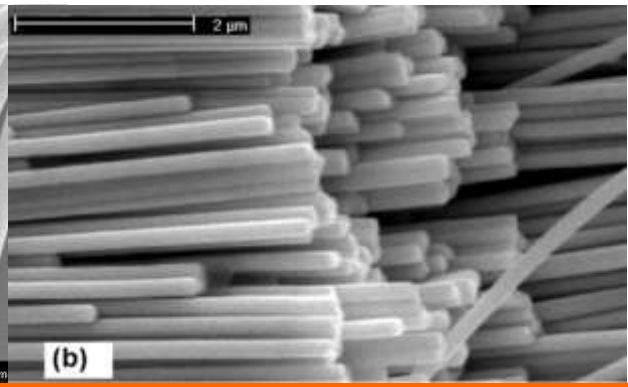
M.E. Toimil-Molares

Materials Research Department
GSI Helmholtz Center for Heavy Ion Research
Darmstadt (Germany)









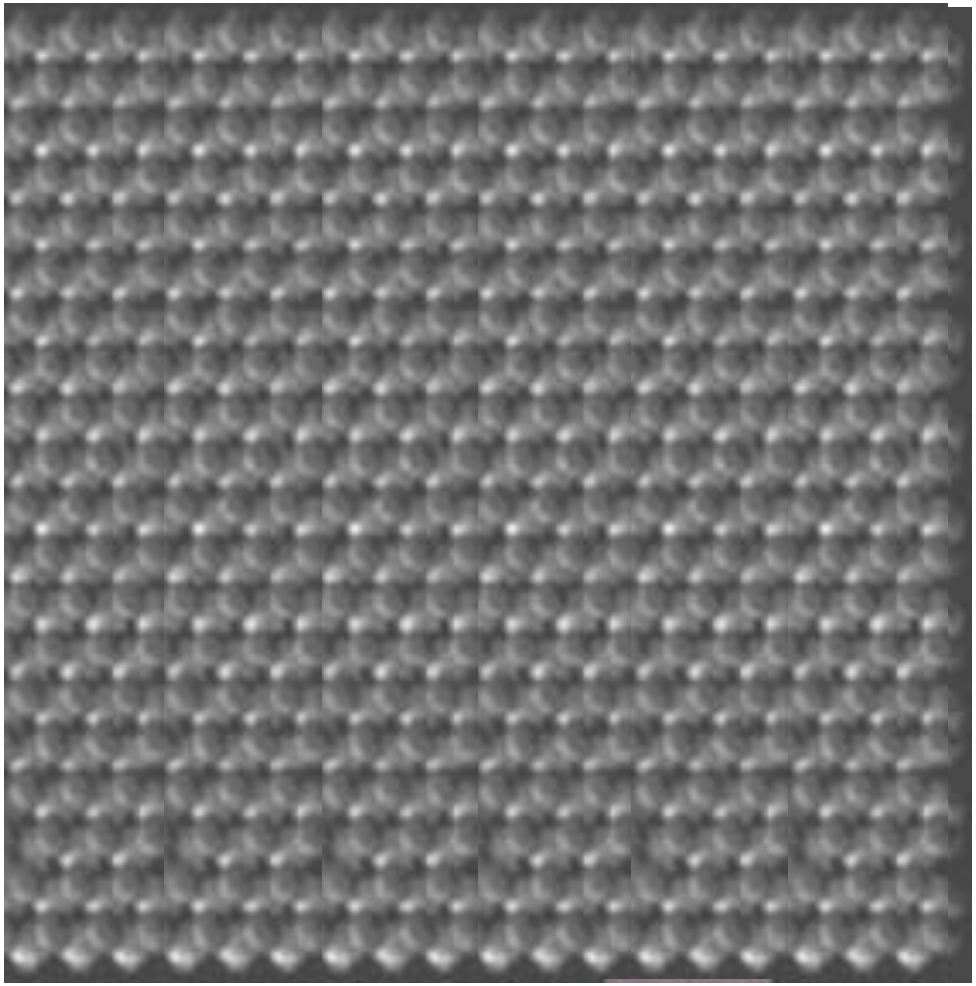
Outline

- What is the template method?
- Which kind of structures can we fabricate?
- Which parameters can we control and to which extent? e.g. material, geometry, size, crystallinity
- Examples of micro- and nanostructures currently developed at GSI

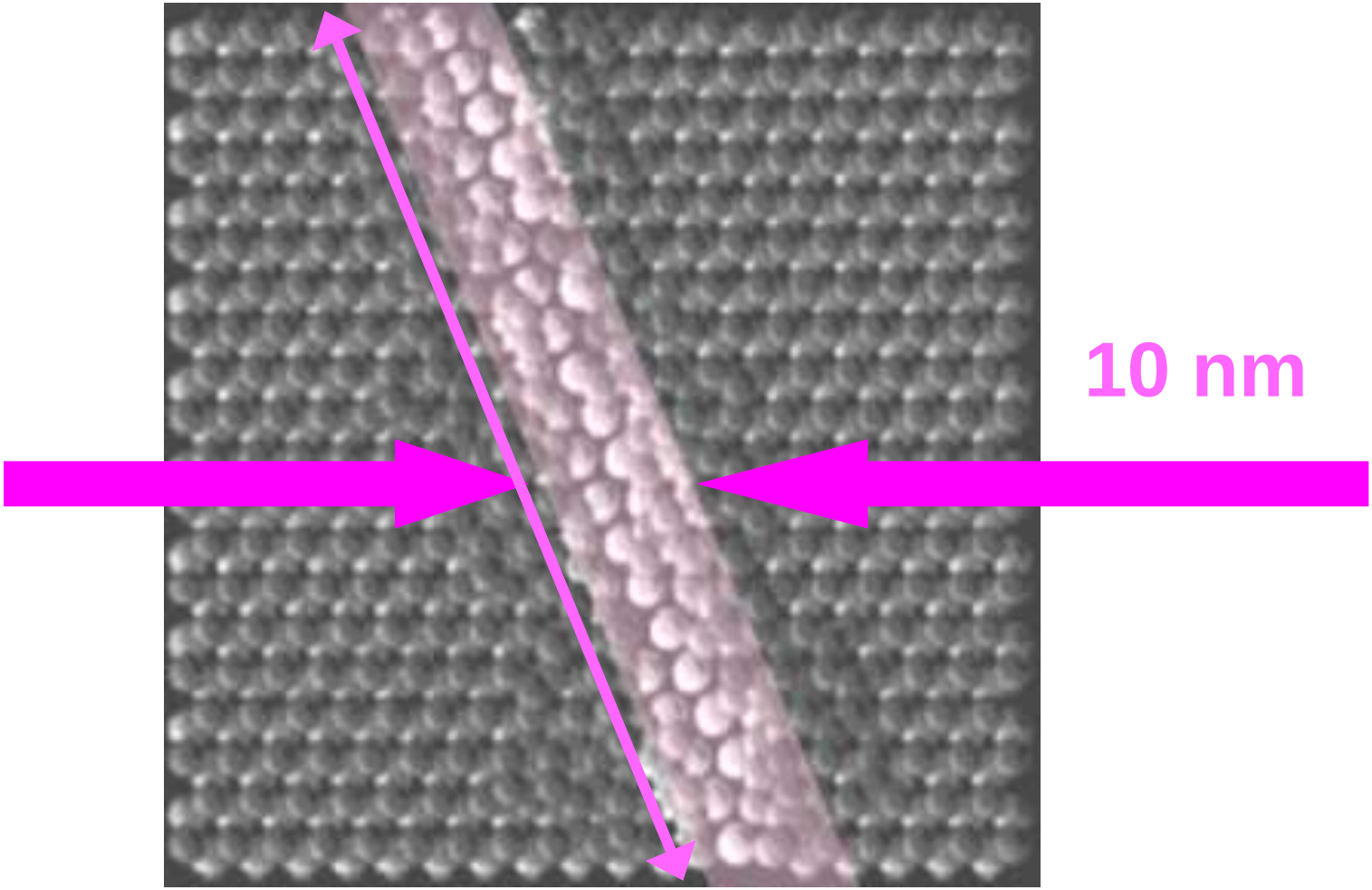
ion beams

kinetic energy: MeV- GeV

10% of velocity of light

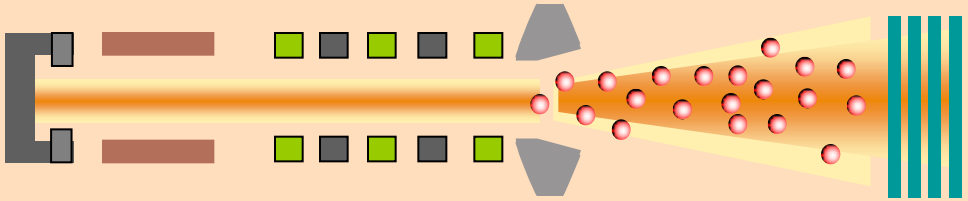


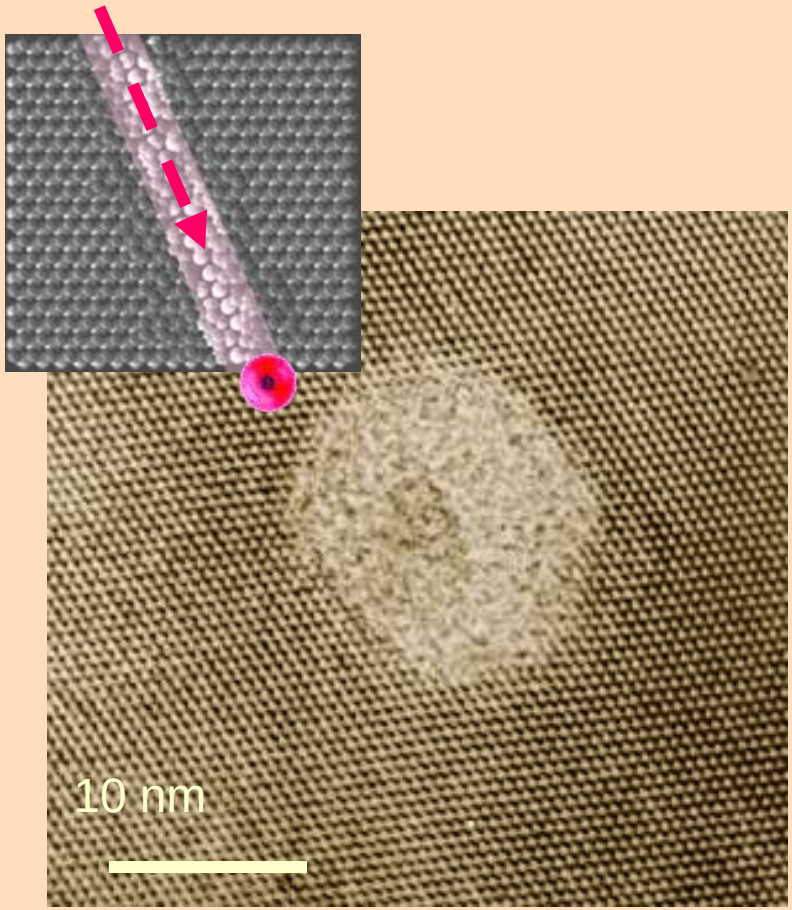
ion track = nanostructure

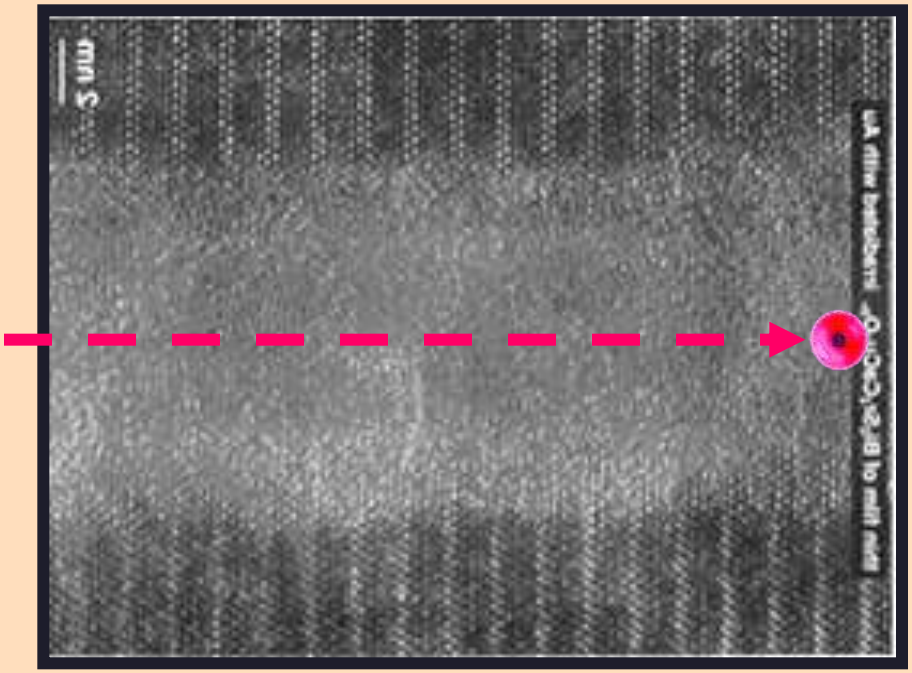


Nanostructures by Heavy Ion Lithography

- Energy (max.) ~ 2 GeV
- Range: ~ 100 µm
- Fluence: 1 - 10^{10} ions/cm^2

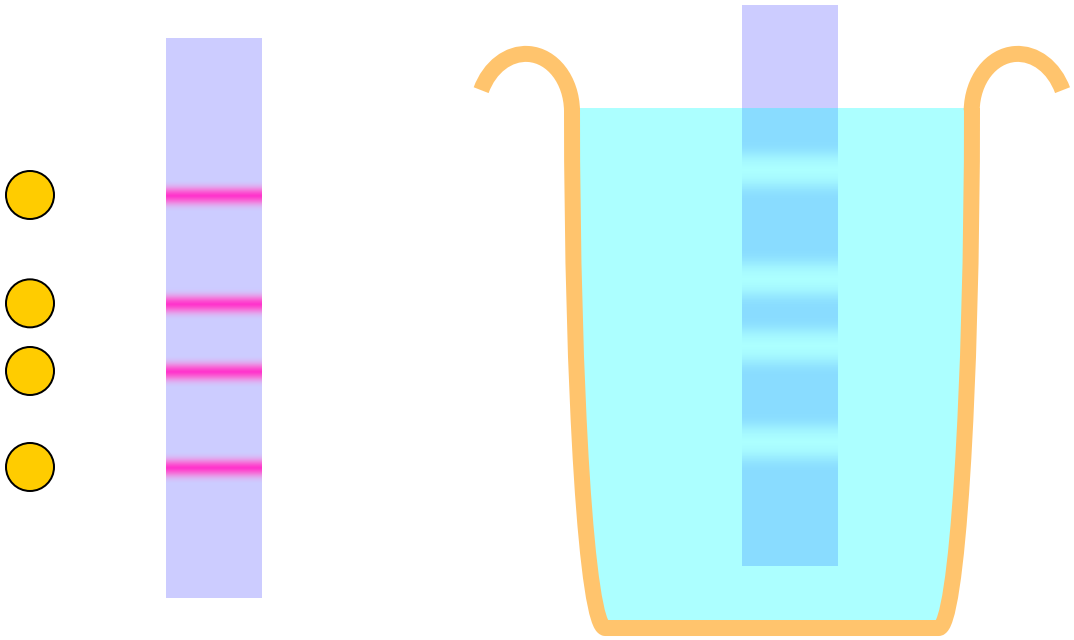






Latent ion tracks

Fabrication of micro- and nanopores



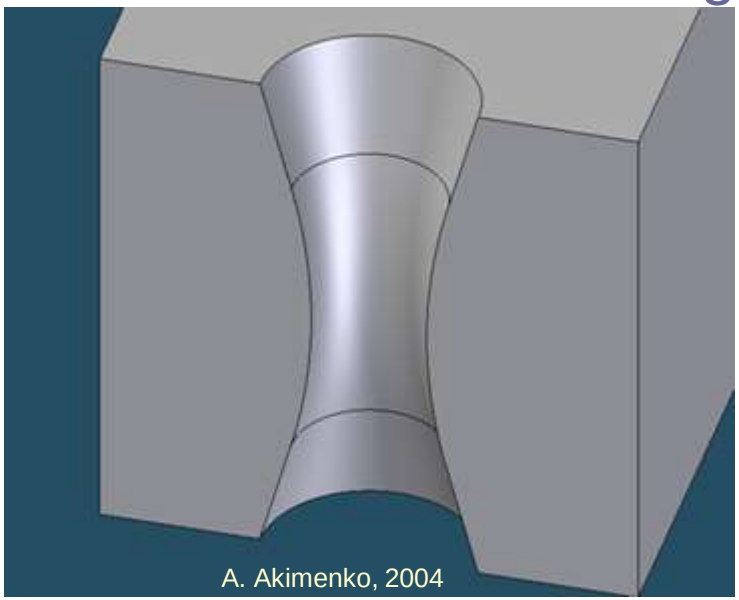
irradiation

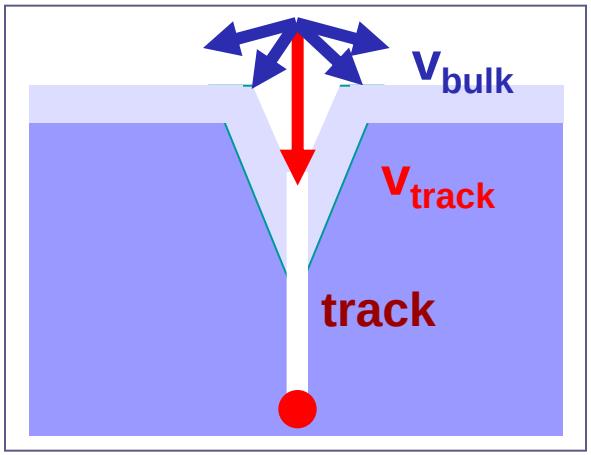
Polymers
Mica
SiN thin films

chemical etching

(NaOH, NaOCl. HF)

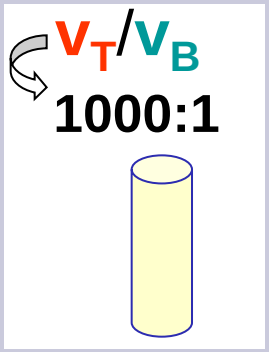
selective ion-track etching

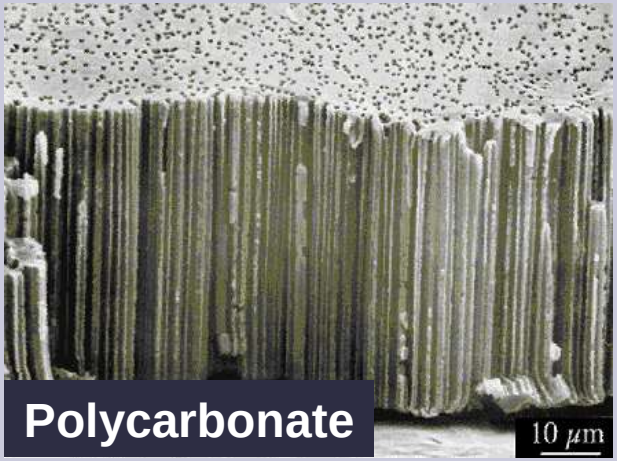


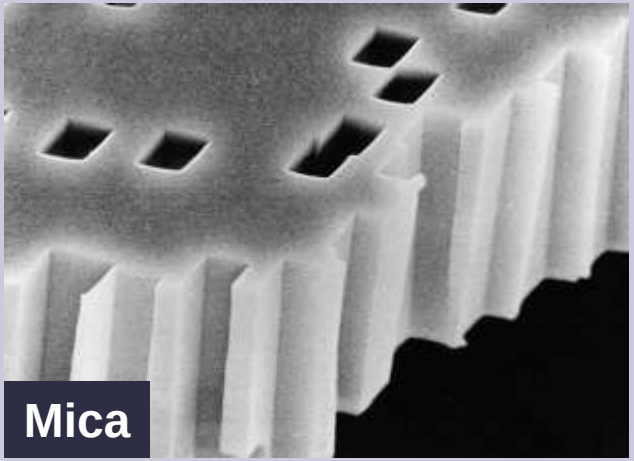


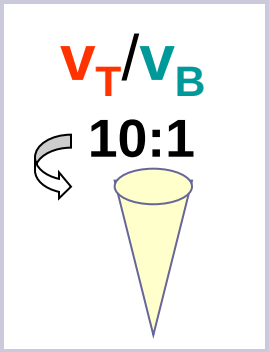
Pore Geometry

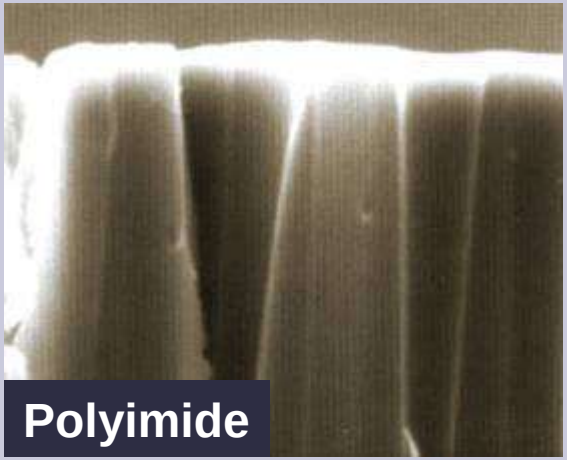
V_{Track} / V_{Bulk} = determines opening angle

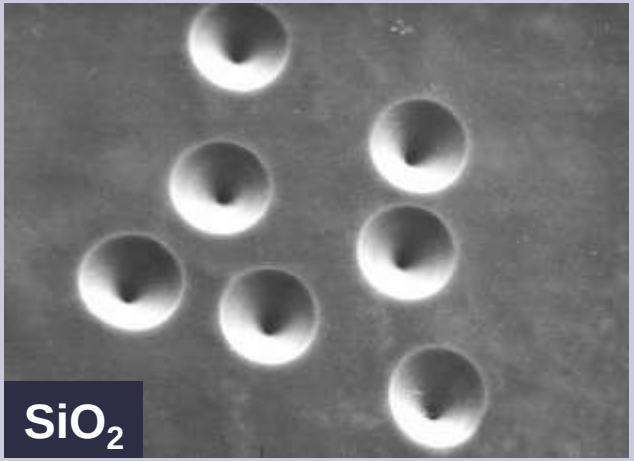




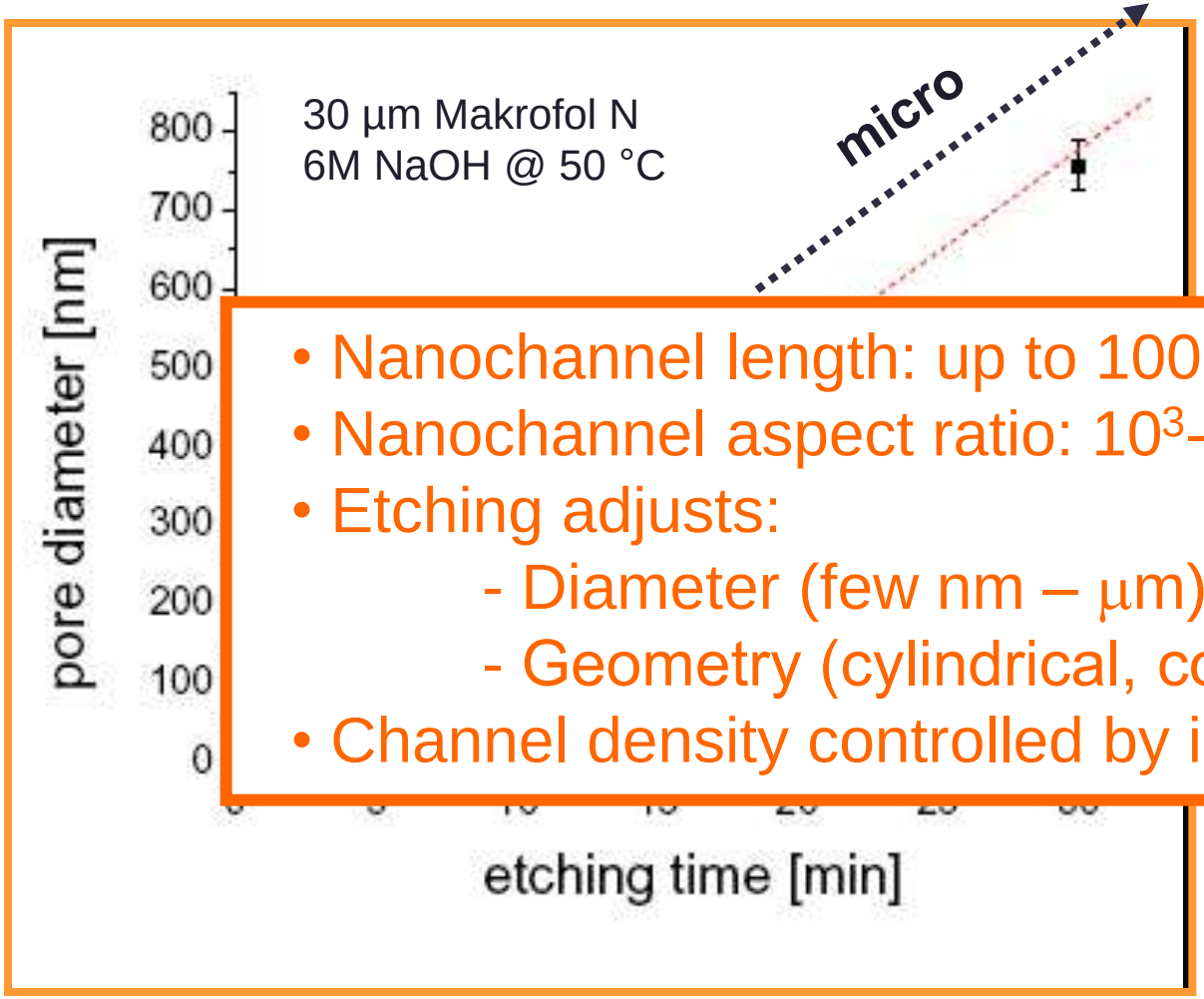


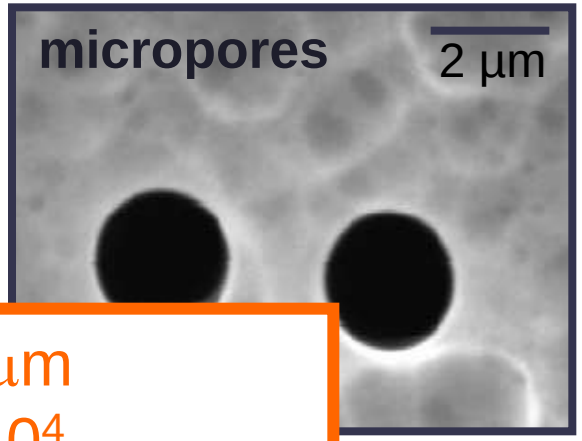




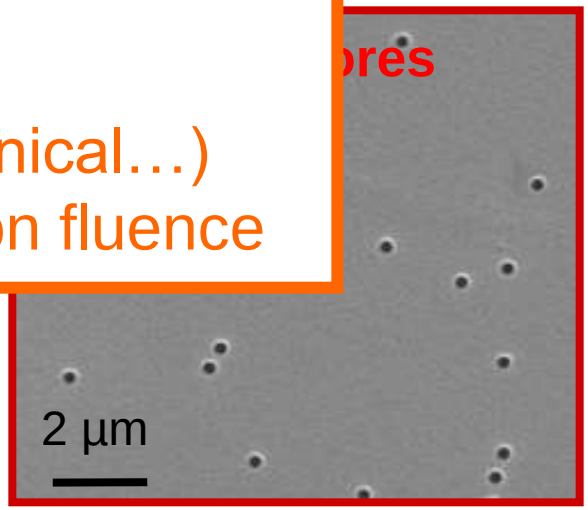


Pore Diameter vs. Etching Time

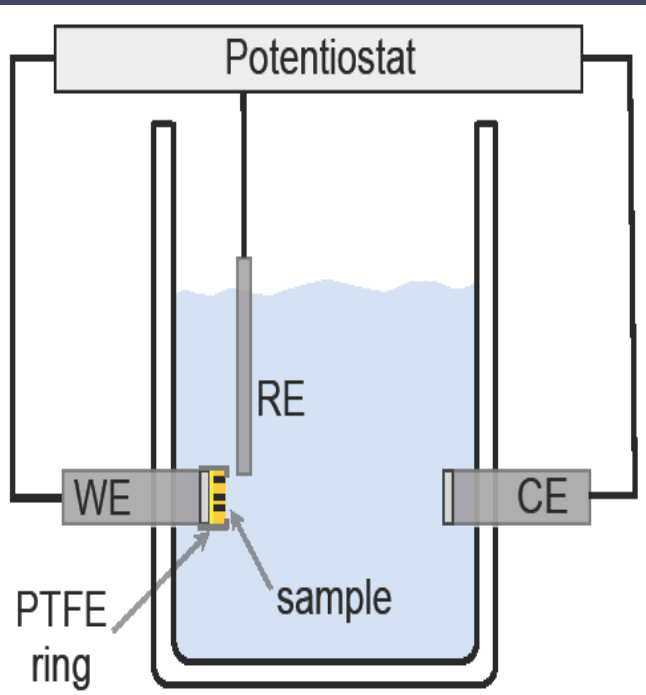


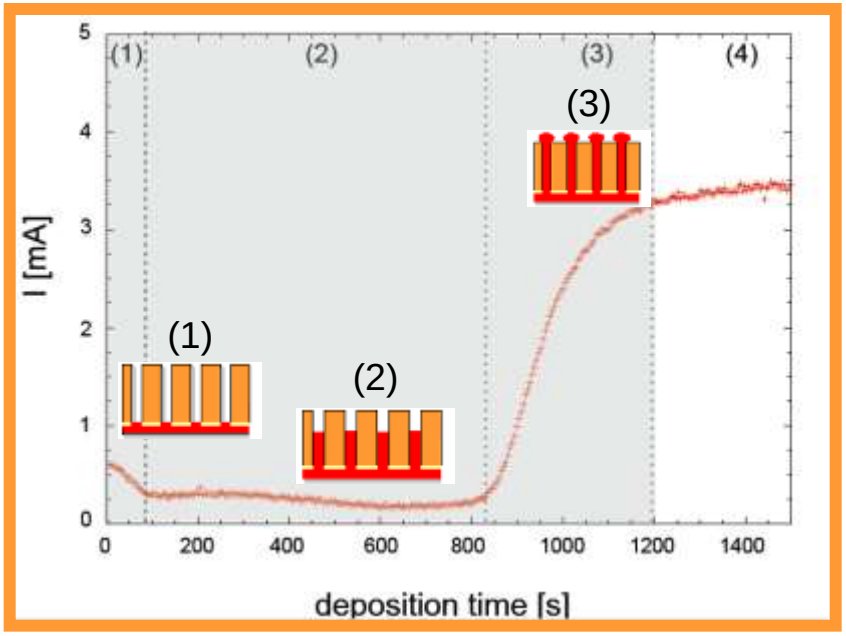


- Nanochannel length: up to 100 µm
- Nanochannel aspect ratio: 10^3-10^4
- Etching adjusts:
  - Diameter (few nm – µm)
  - Geometry (cylindrical, conical…)
- Channel density controlled by ion fluence



Electrodeposition of Micro- and Nanowires





Parameters:

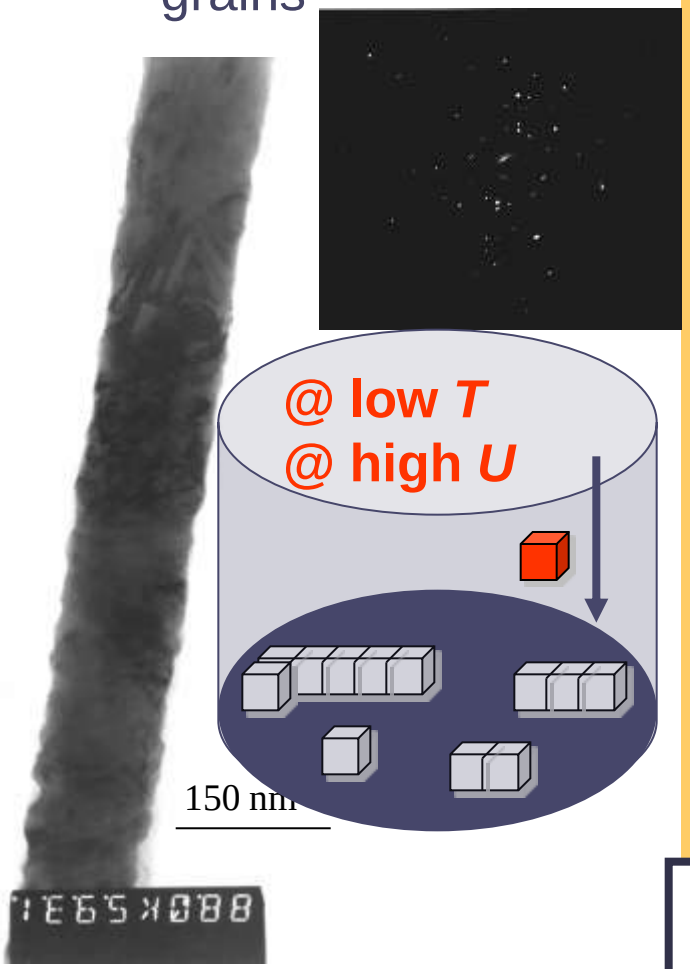
Electrolyte
Temperature
Voltage
Cell geometry
Convection
(stirring, ultrasound)
Electrodes

Control:

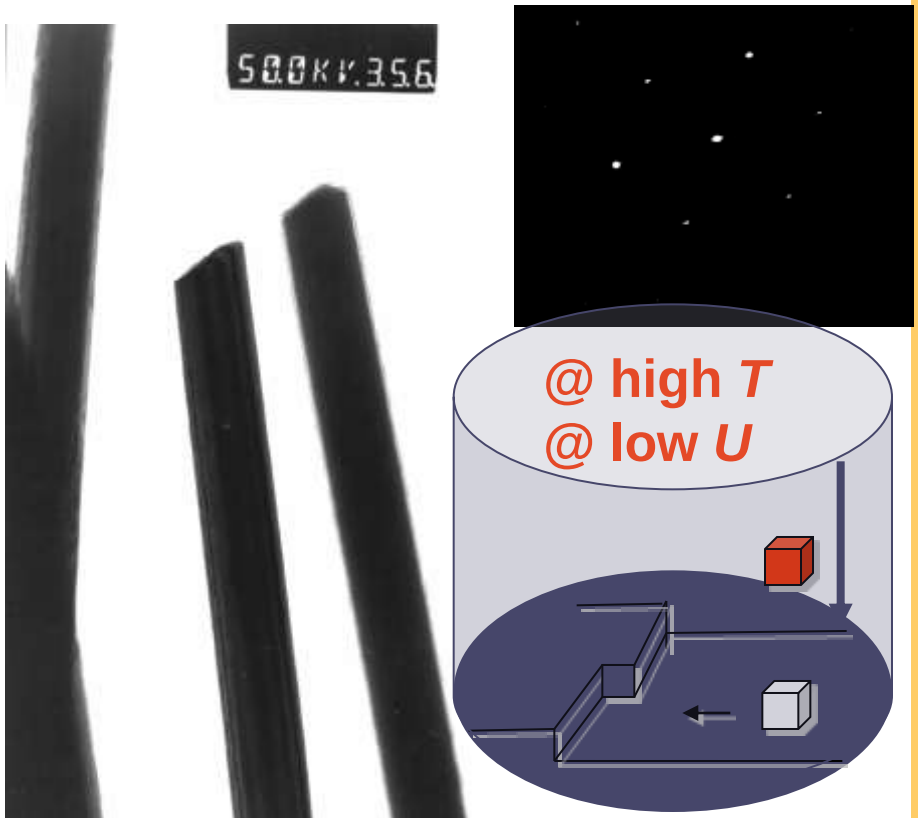
Composition
Crystallinity
Roughness

Control over Crystallinity

Nucleation and formation of new grains



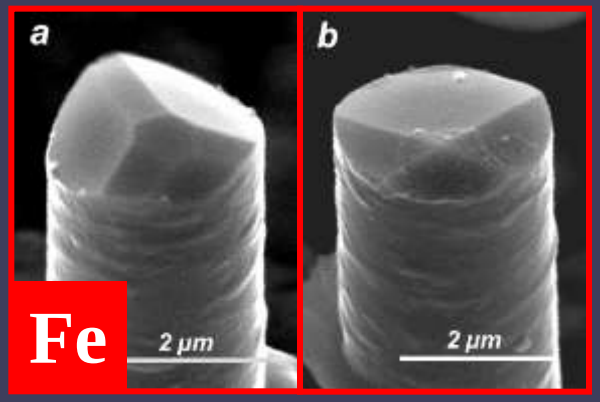
Build up of existing crystals

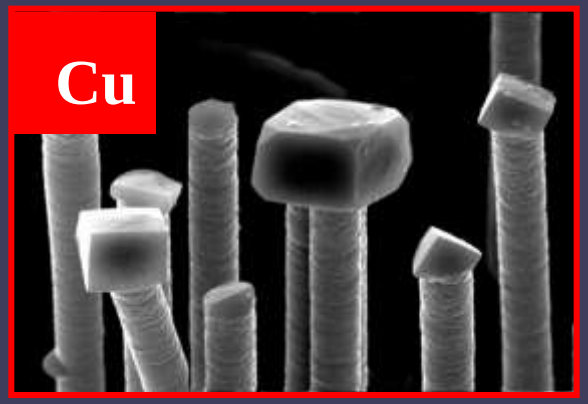


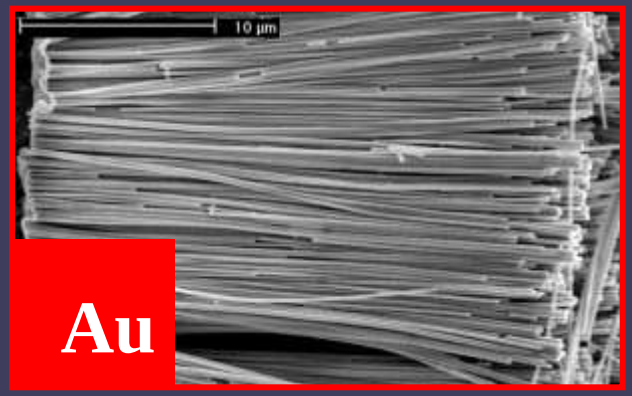
- Copper nanowires
- Cu_2SO_4 based electrolyte
- 23 – 50 ºC
- Potentiostatic

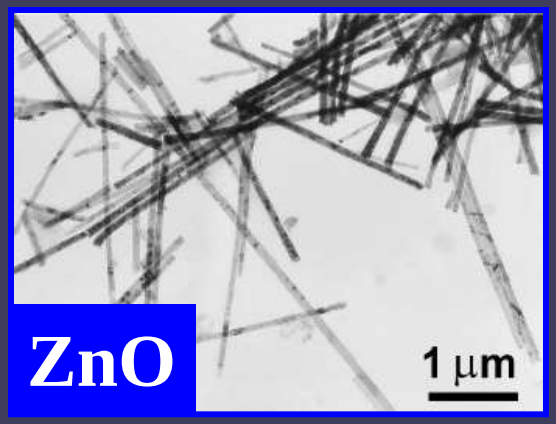
M.E. Toimil-Molares et al. Adv. Mater. (2001)

Materials Research









Metals: Cu, Au, Ni, Fe, Pt

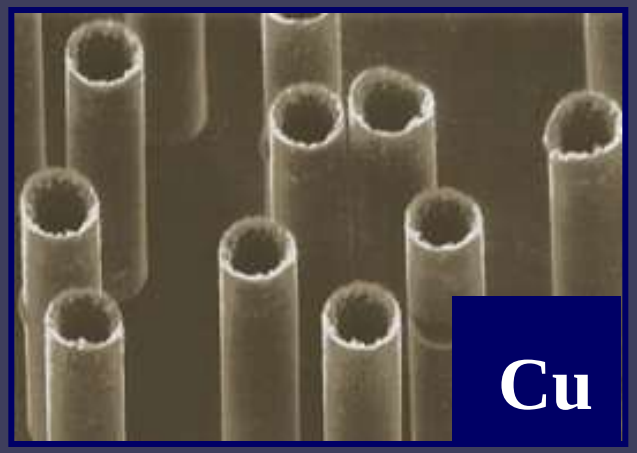
Semimetals: Bi

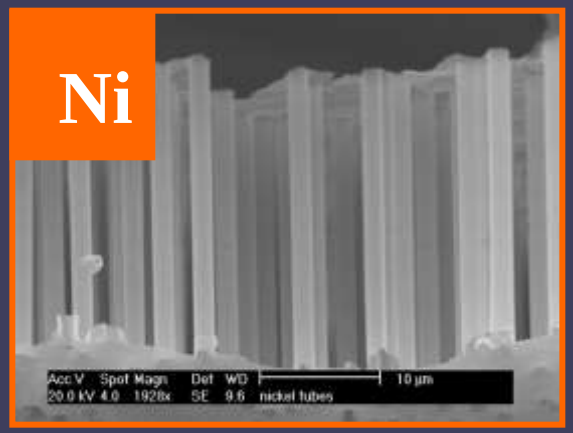
Semiconductors: ZnO

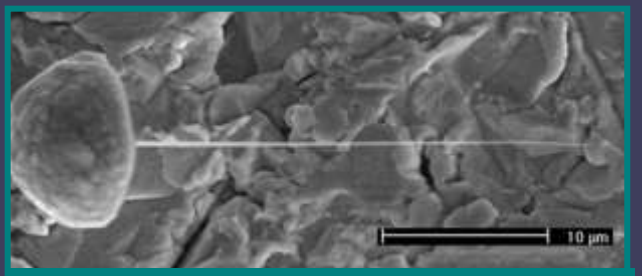
Multilayers: Cu/Co

Choice of electrodeposition conditions → poly- and single-crystalline nanowires:

Cu, Au, Bi, Sb, Fe, Ni, Ag,...



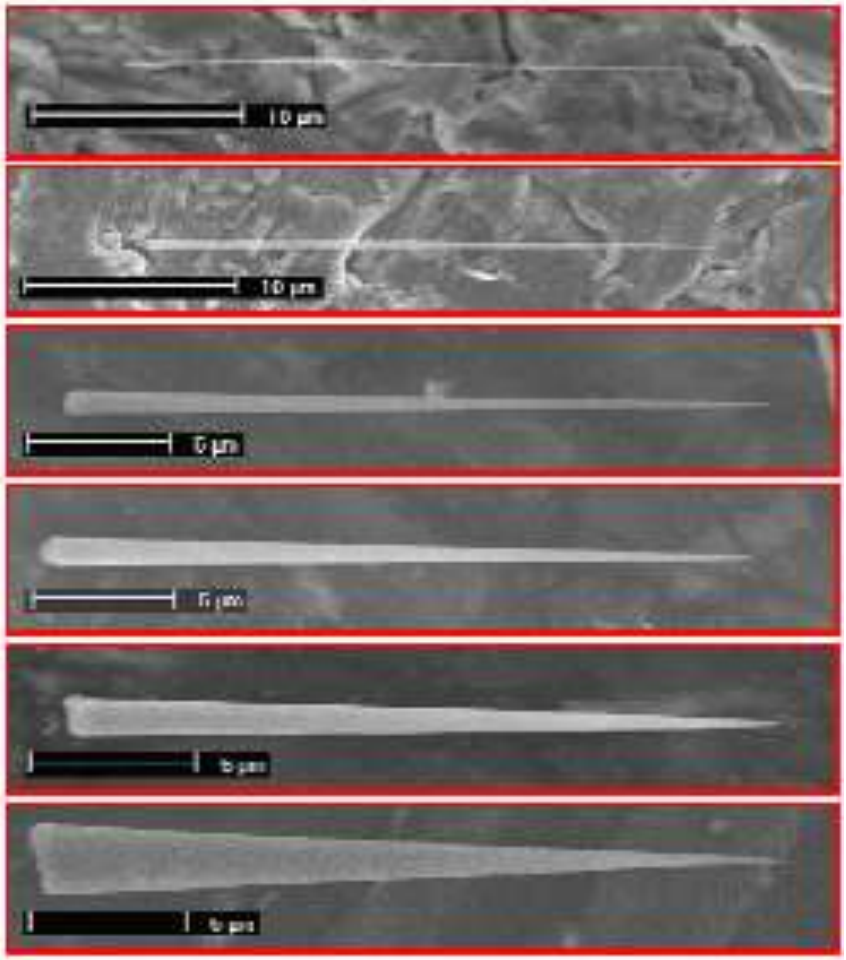




Conical Cu/Co

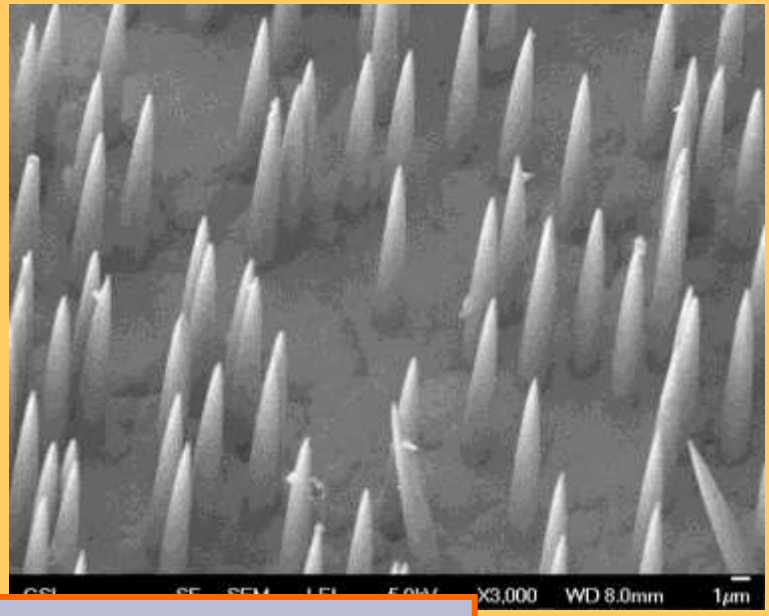
Control over Geometry

Nanowire geometry controlled by etching conditions



Increasing methanol concentration increases the apex angle of the nanostructures

Freestanding conical wires

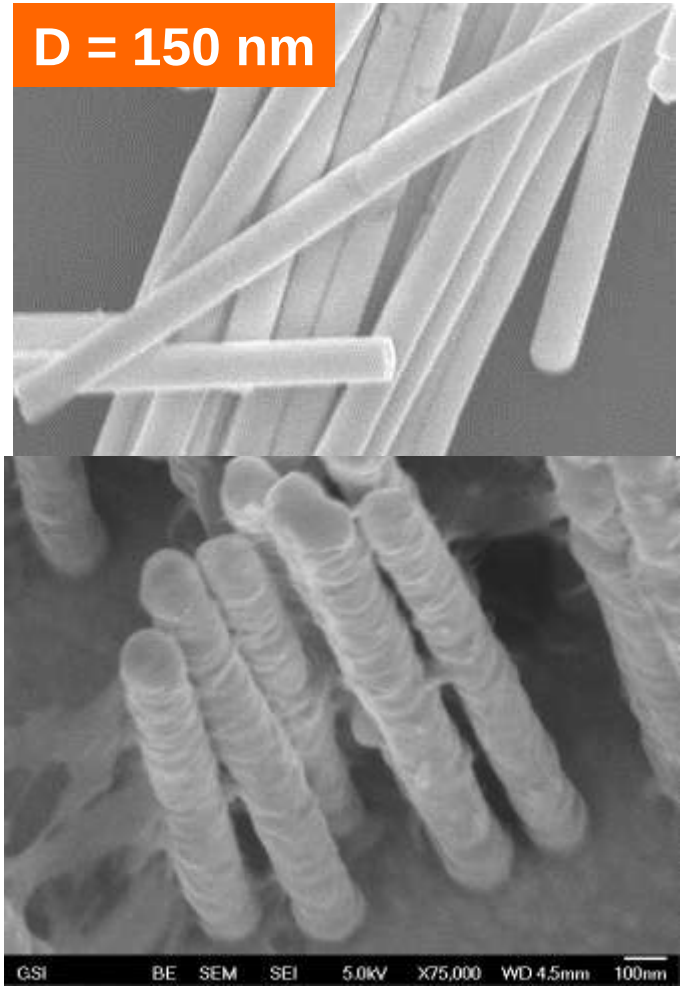


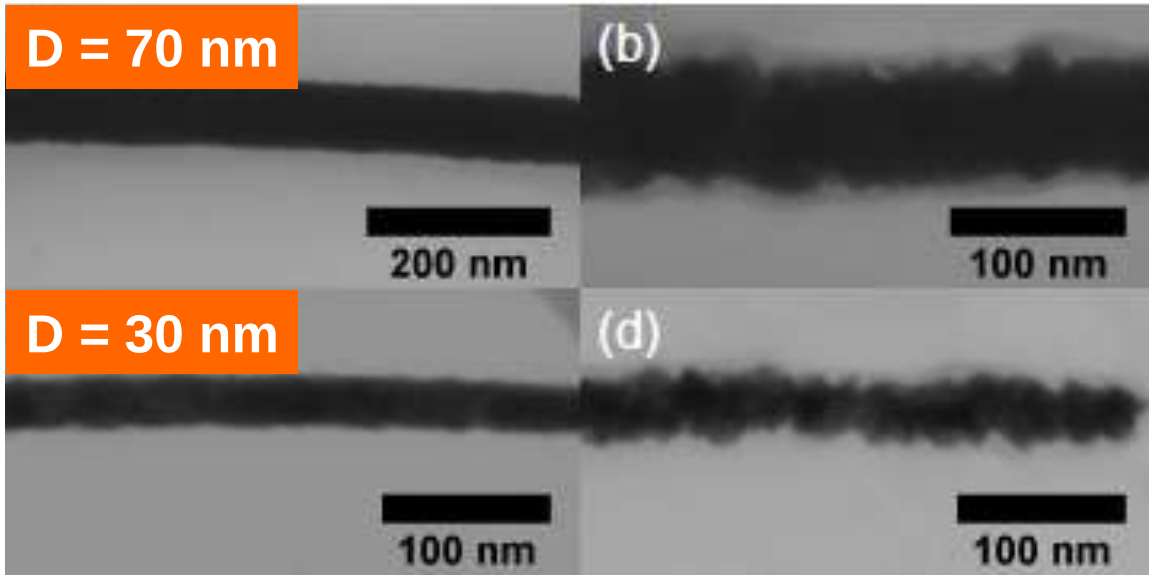
Smooth contour

J.L. Duan (2008), J. Krieg (2010)

Nanowires with various surface roughness

Nanowire morphology controlled by the inner surface of the nanopores in the template

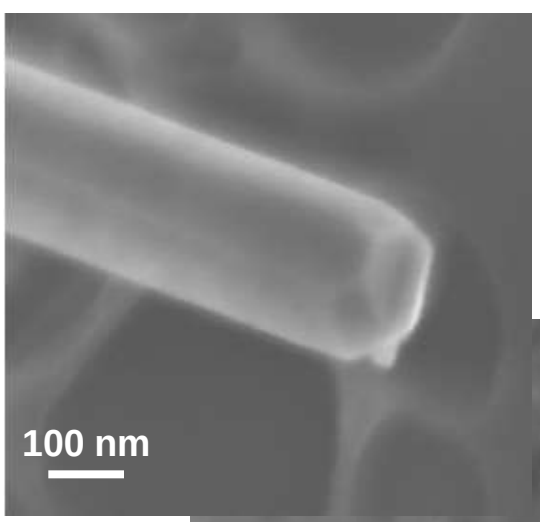




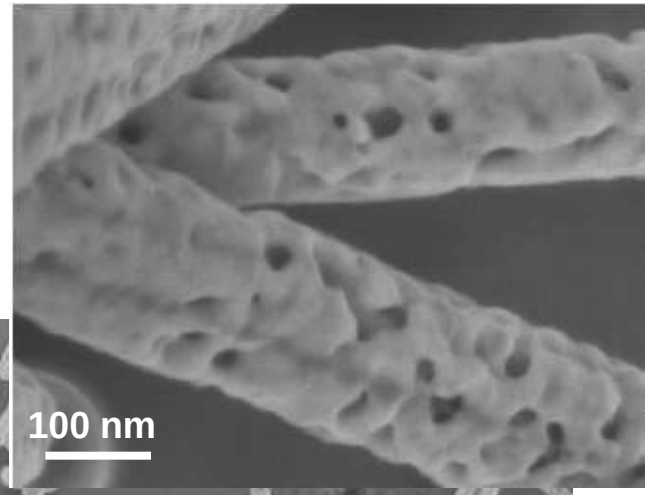
S. Mueller et al. Crystal Growth and Design (2012)

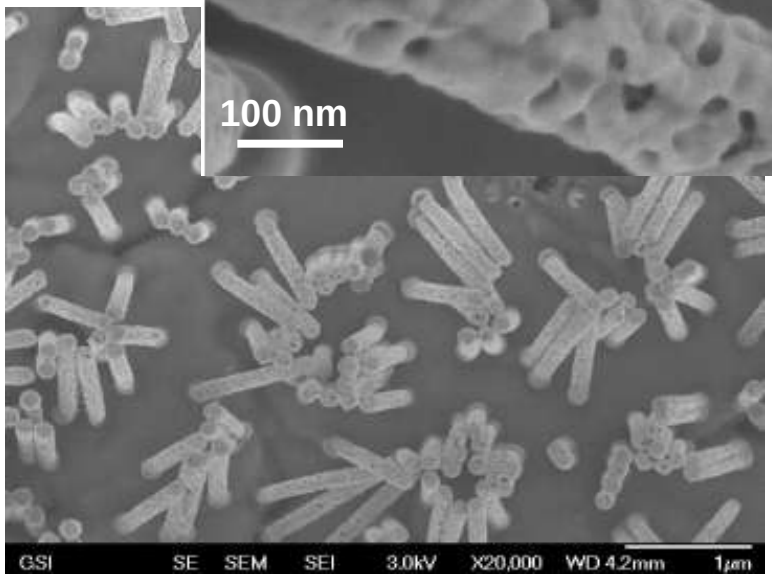
Materials Research

AuAg alloy and porous Au nanowires



Ag dissolution



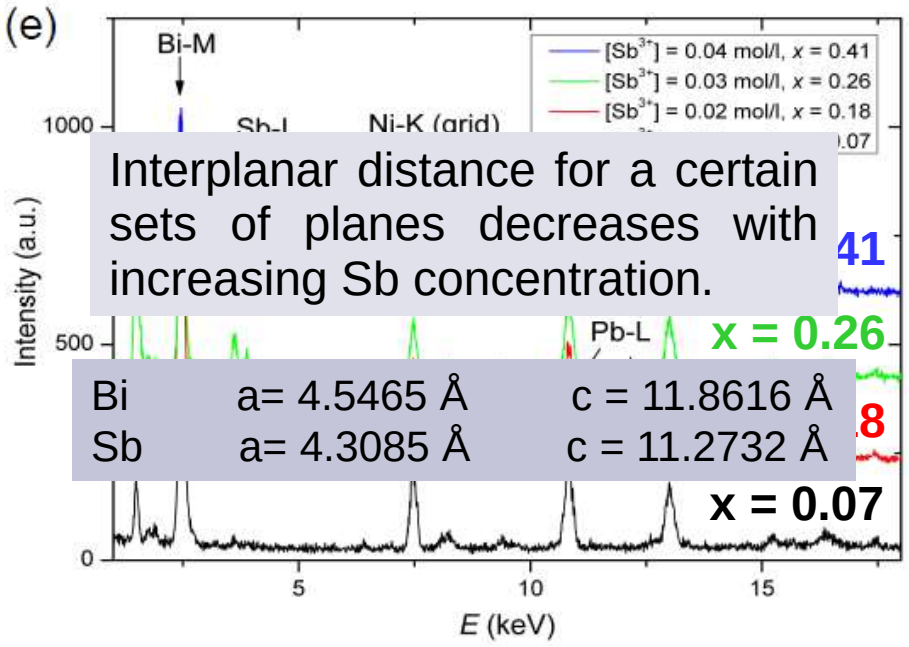


→ Constant *U*
→ Facetted silver gold nanowires indicating large crystals

→ Porous Au wires with 10-30 nm pores
→ Reduced diameter after Ag etching

Composition of $Bi_{1-x}Sb_x$ nanowires

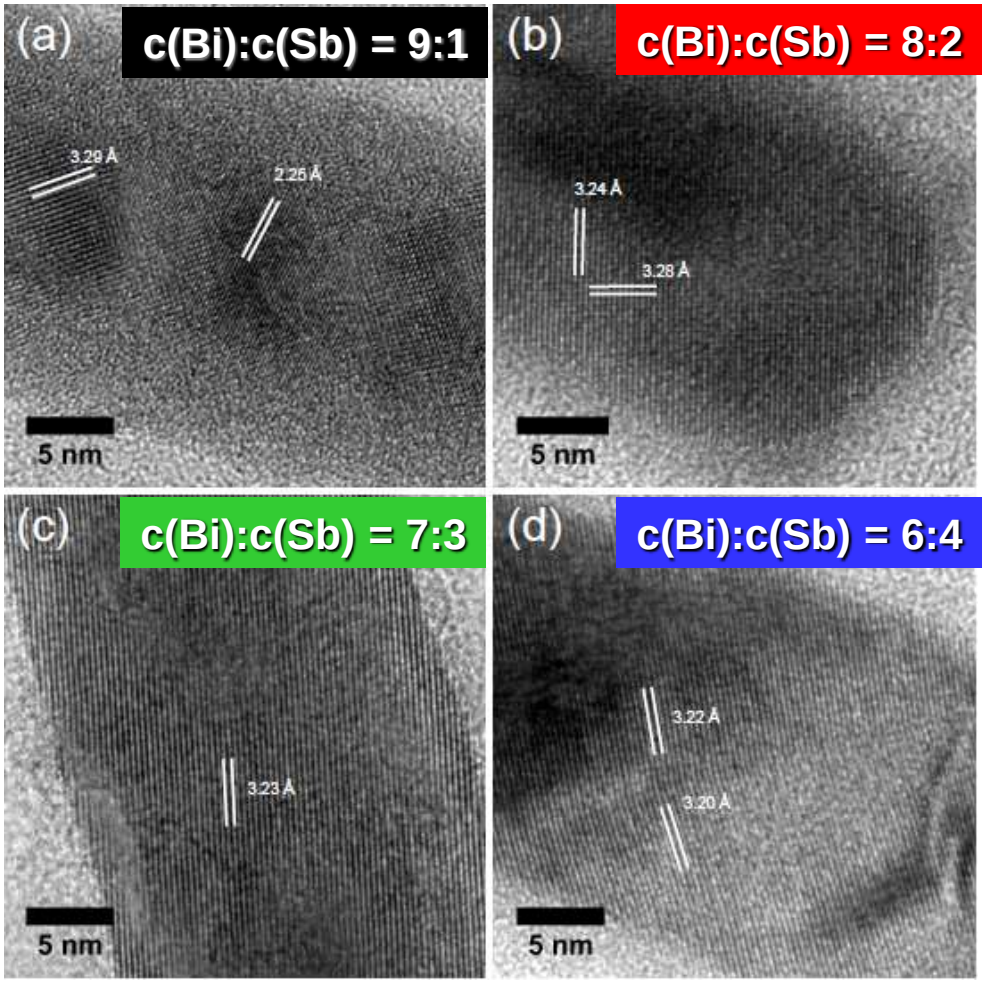
HRTEM-EDS



Interplanar distance for a certain sets of planes decreases with increasing Sb concentration.

Bi	a= 4.5465 Å	c = 11.8616 Å
Sb	a= 4.3085 Å	c = 11.2732 Å

An increase in the concentration of Sb ions in the electrolyte results in an increase in Sb concentration in the alloy wires.

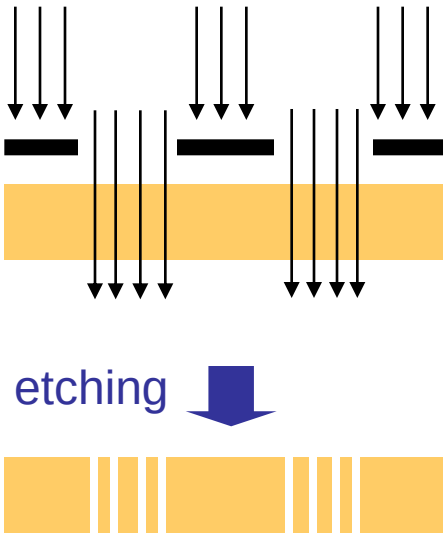


S. Mueller *et al*. Crystal Growth and Design (2012)

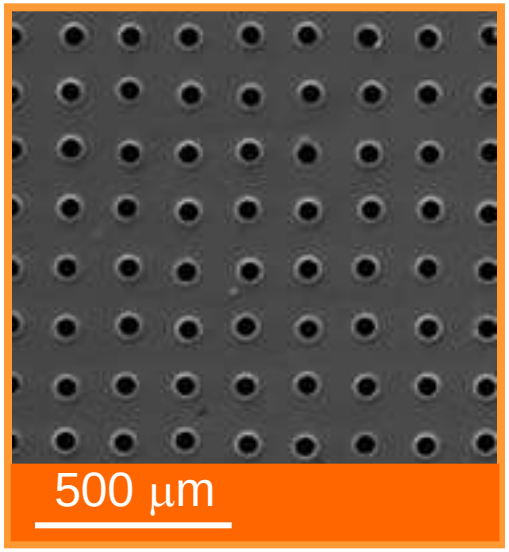
GSI Materials Research

Ion Track Technology: to close the nano-micro gap

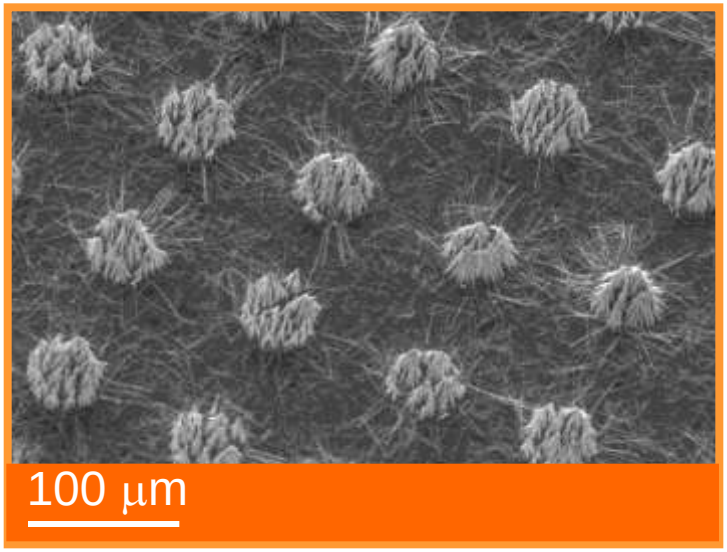
1. Irradiation through mask to microstructure etched ion track membranes →



mask

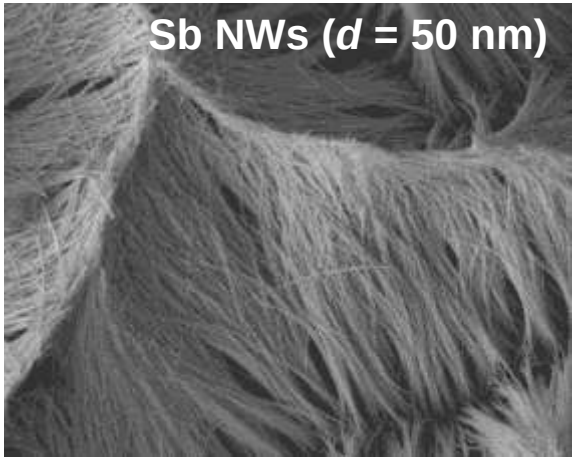


50 μm diameter NW arrays

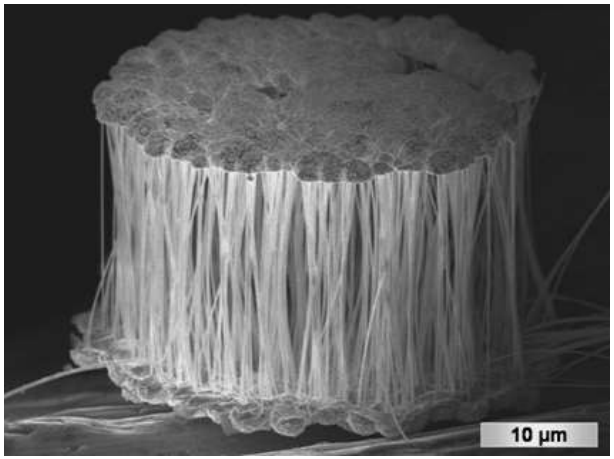


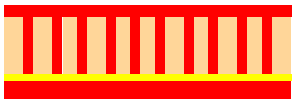
Ion Track Technology to close the nano-micro gap

2. To remove membrane maintaining mechanical stability of the NWs.

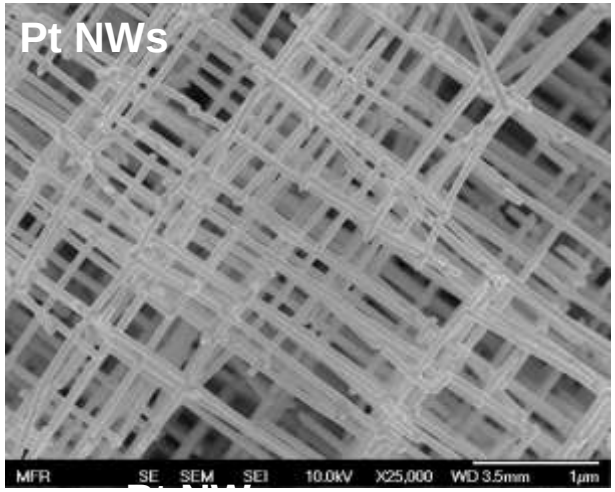


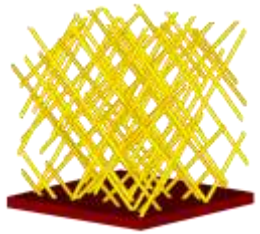
Typical nanowire "spaguetti" after template dissolution





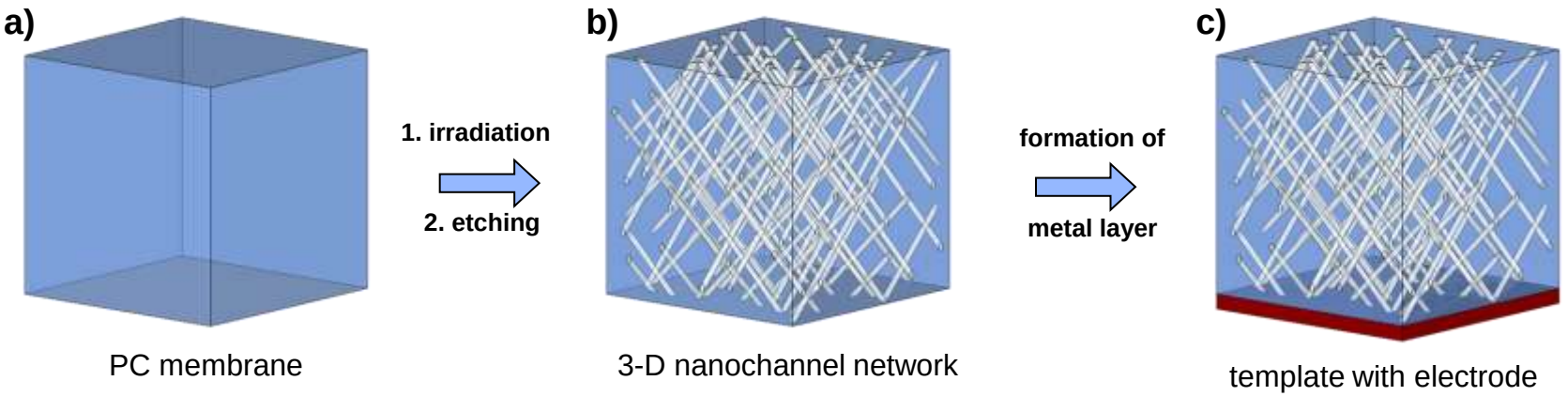
NW „microreactor“





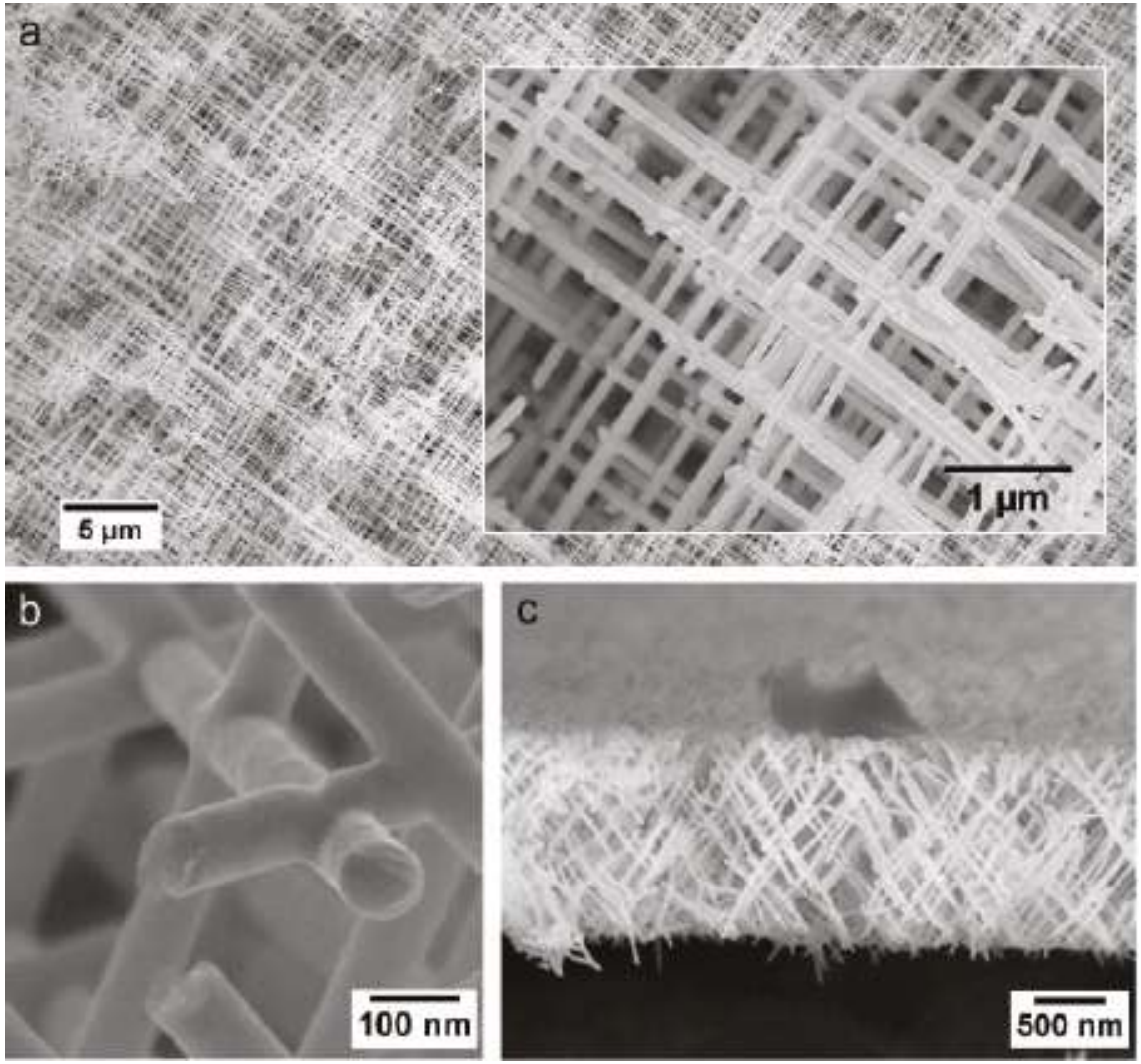
NW Network

Ion Track Technology to close the nano-micro gap



Ion Track Technology to close the nano-micro gap

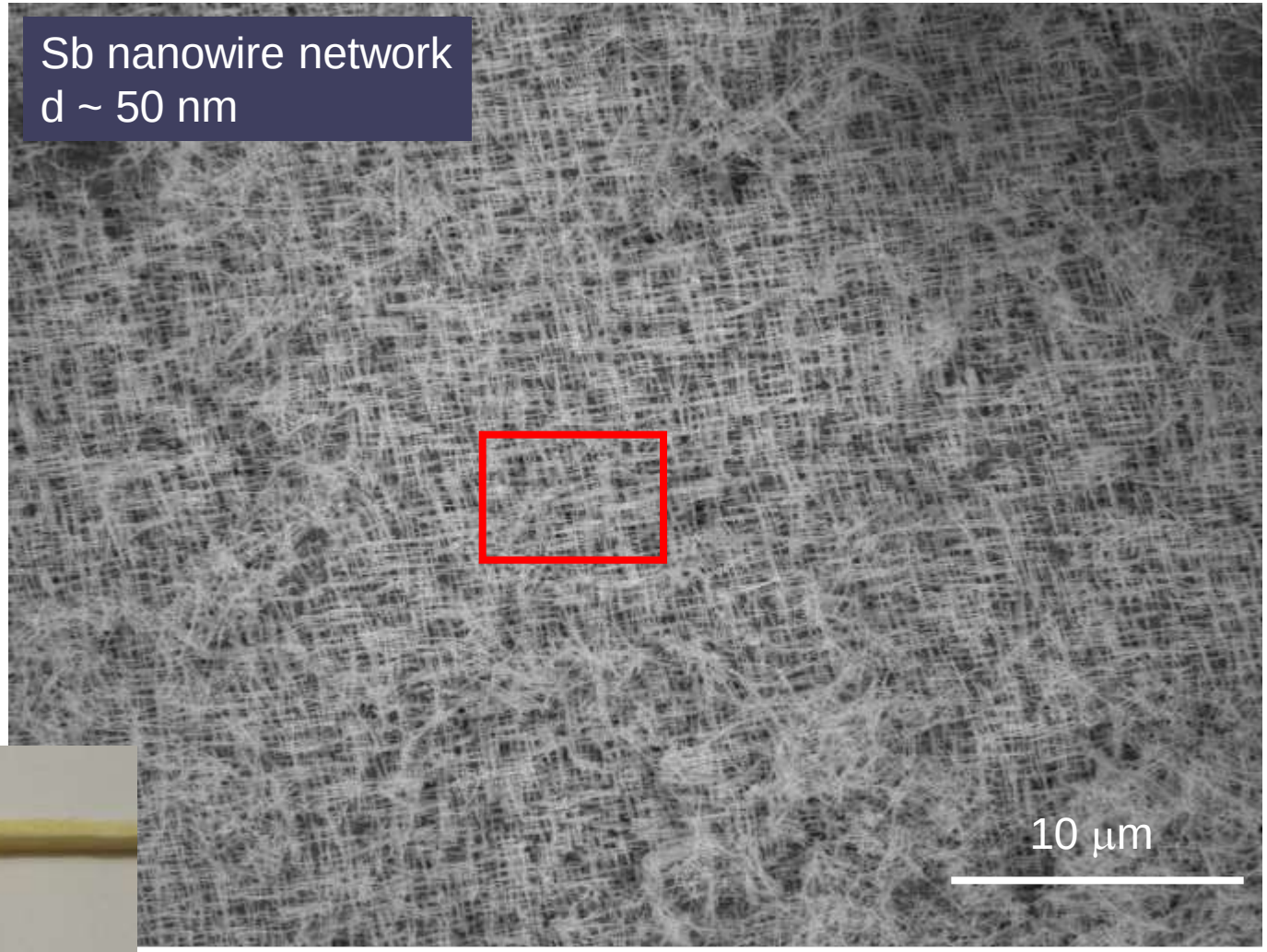
Pt



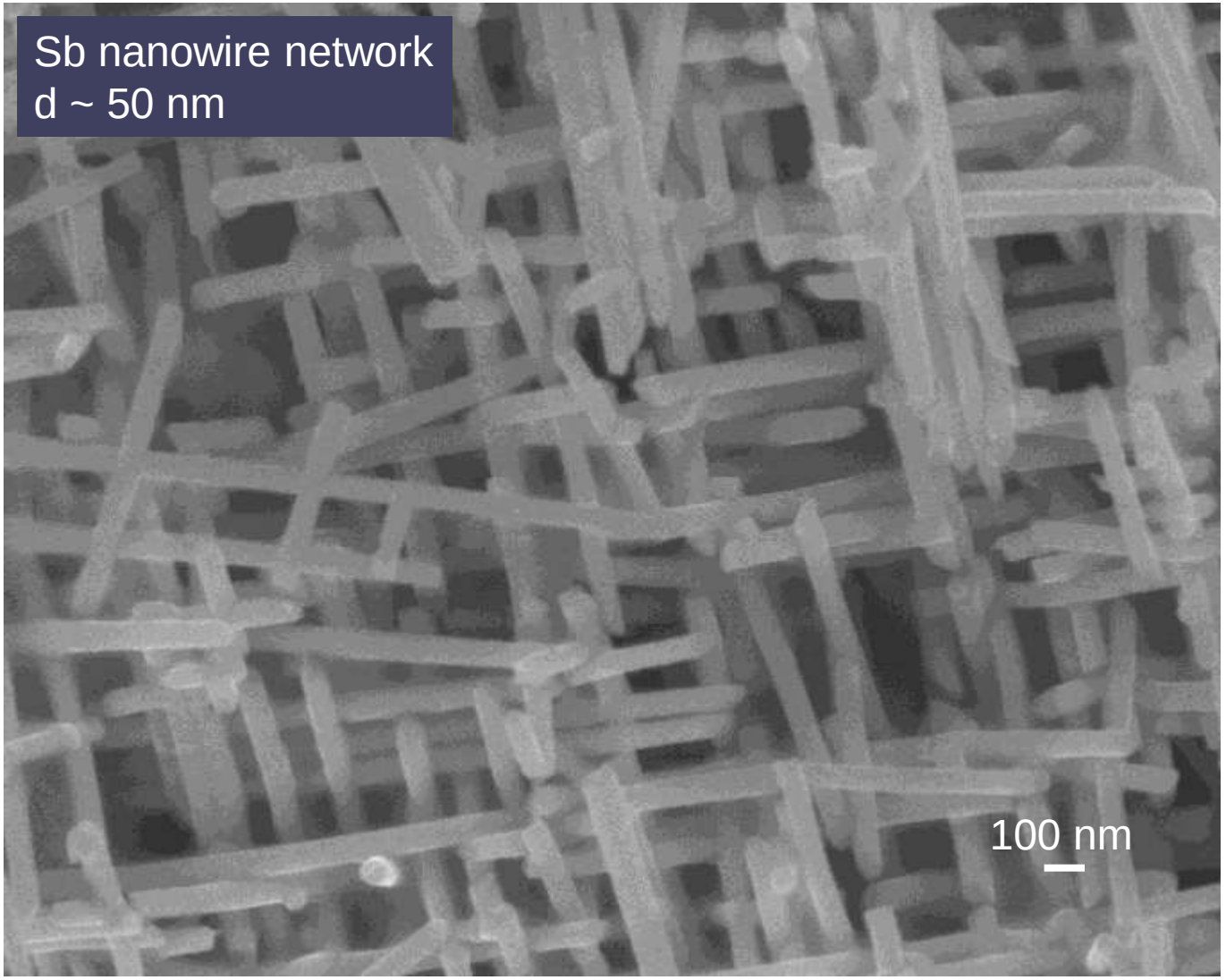
Rauber et al. Nano Lett. 11, 2011, 2304

3D Nanowire Networks

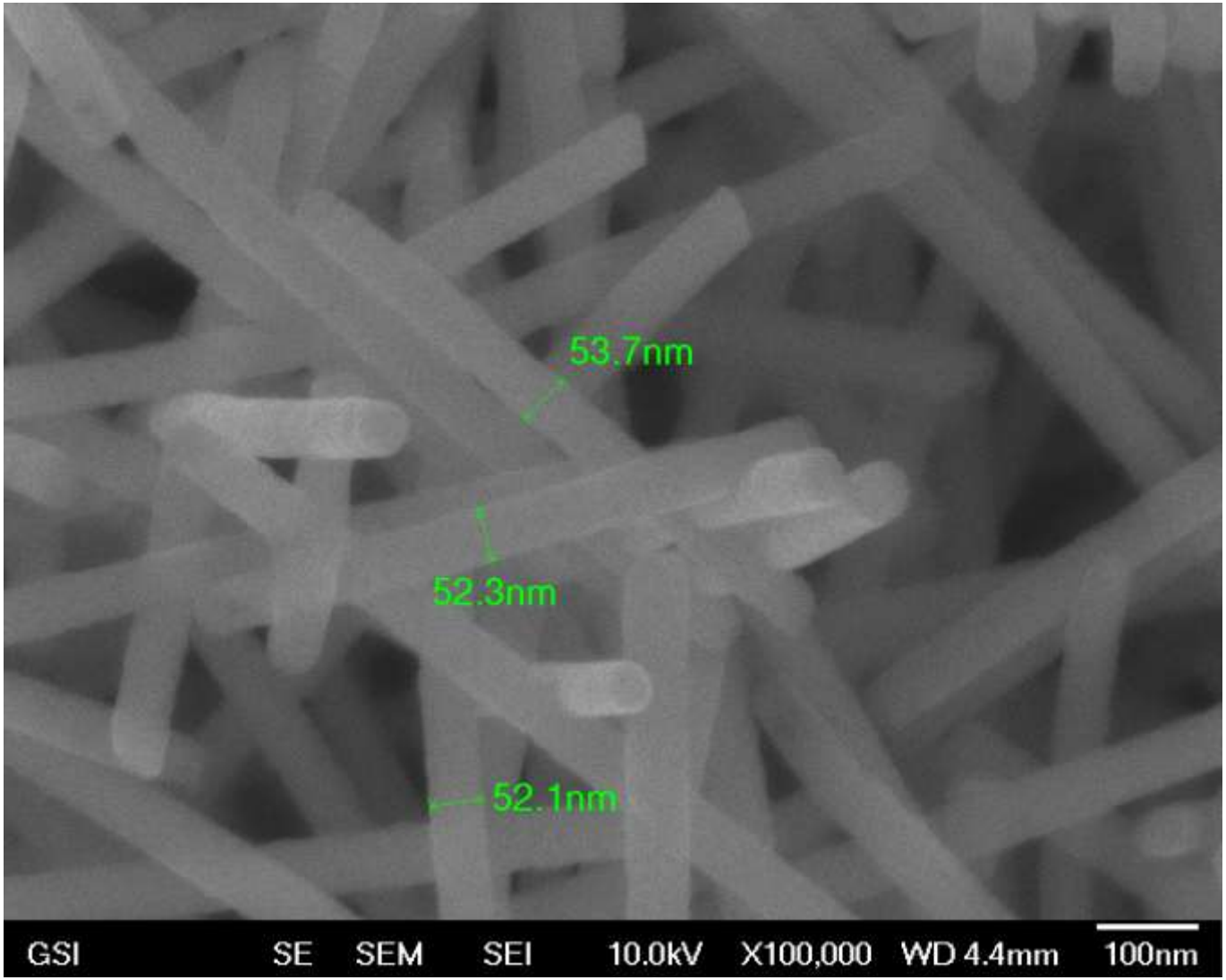
Sb



3D Nanowire Networks

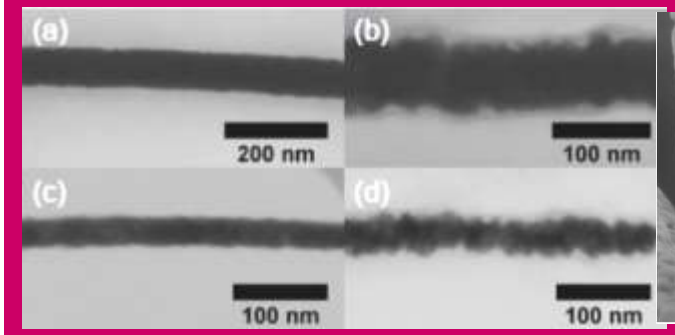


3D Nanowire Networks

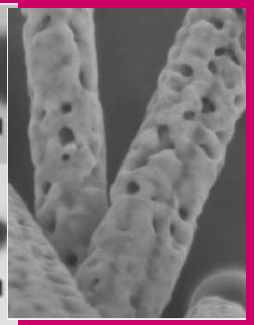


Micro- and Nanostructures *Palette*

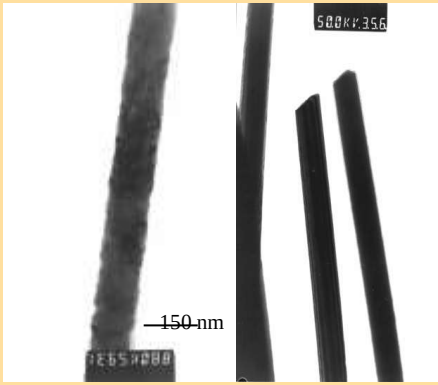
Smooth vs rough



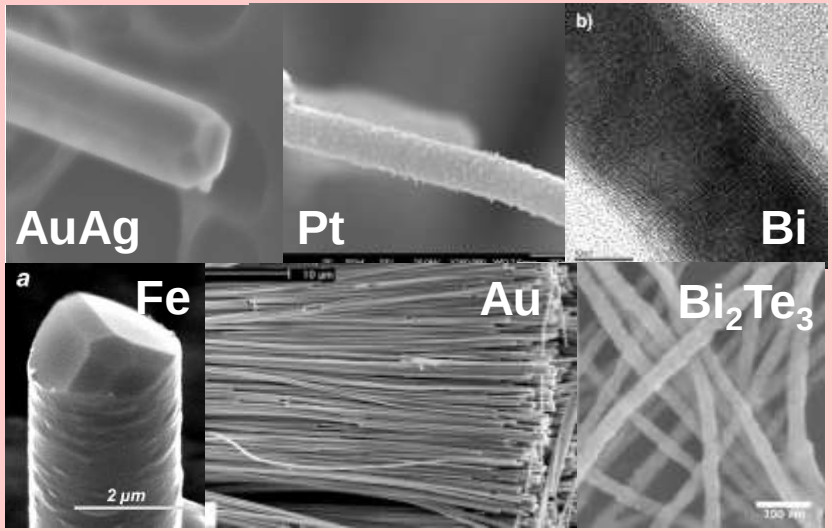
Porous



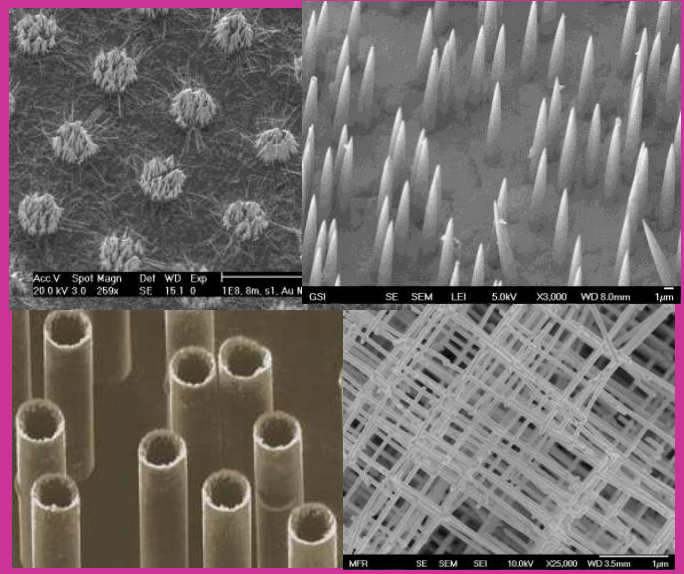
Polycrystalline and single-crystalline



Materials



Tubes, cones, networks…



Conclusions

→ **A large variety of micro- and nanowire structures can be fabricated by electrodeposition in etched ion track membranes.**

→ **Excellent control over material, geometry, size, crystallographic properties, and surface characteristics, achieved by proper selection of fabrication parameters.**

→ **Interesting objects include:**

- → **Nanowires with controlled length, radius and aspect ratio**
- → **Smooth, rough and porous nanowires**
- → **Nanowires with controlled roughness**
- → **Micro-structured nanowire arrays**
- → **Nanowire networks**

→ **Investigation of their properties is underway:**

- → **Optical properties (IR-spectroscopy, EELS-STEM)**
- → **Electrical properties**
- → **Thermoelectrical properties**
- → **Thermal stability**

THANKS!!

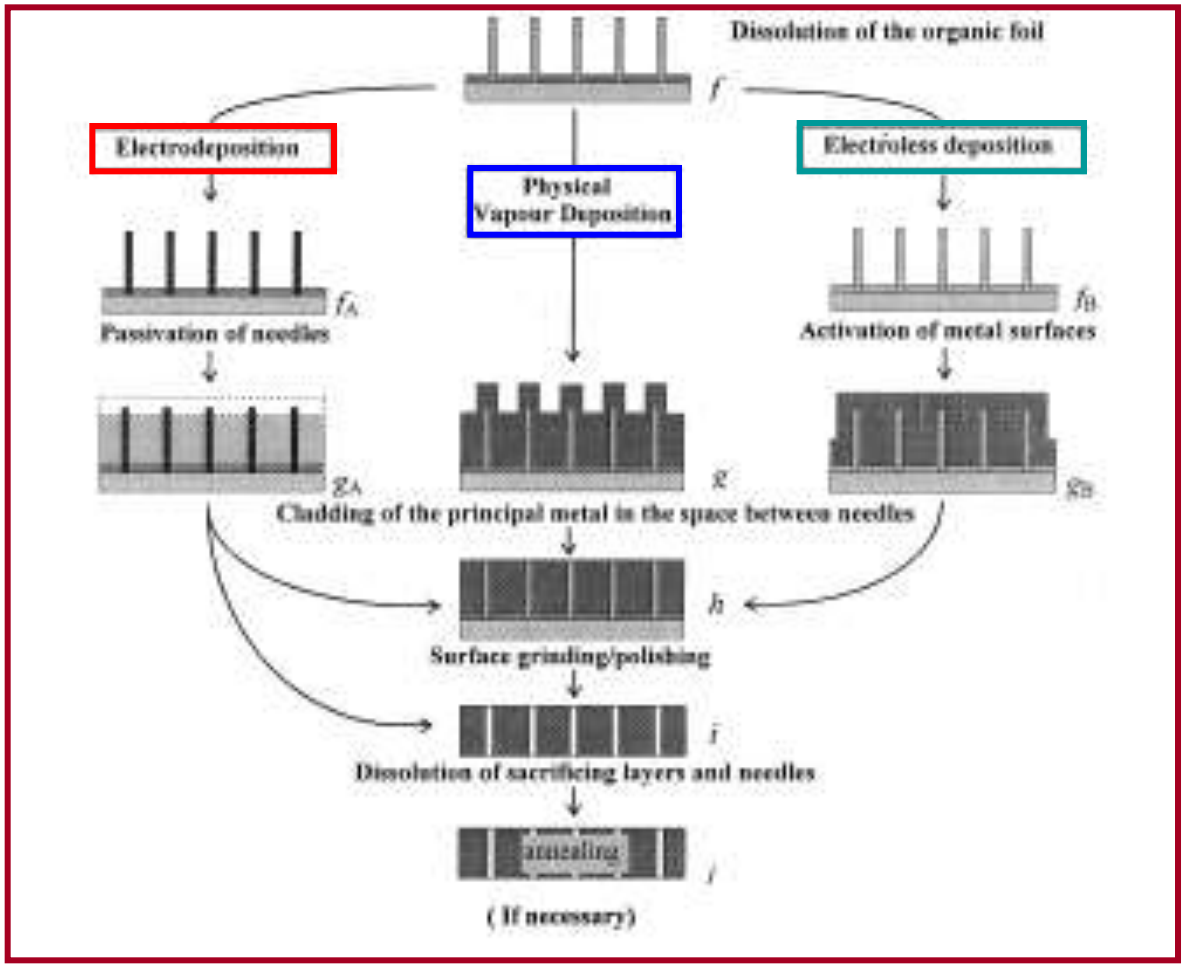
GSI Nanowire Students :

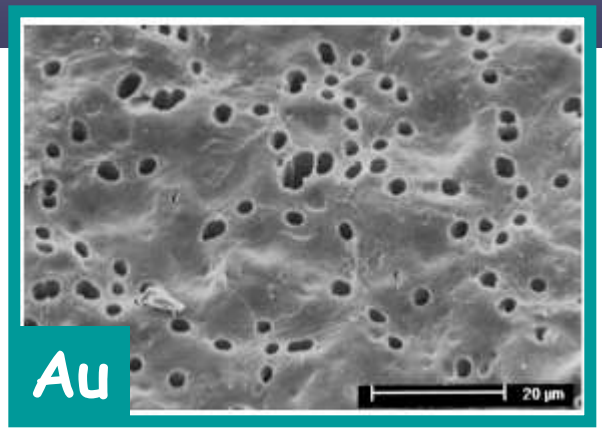
Ina Alber, Sven Mueller, Oliver Picht, Michael Wagner, Markus Rauber, Janina Krieg, Christian Schoetz, Christian Mueller

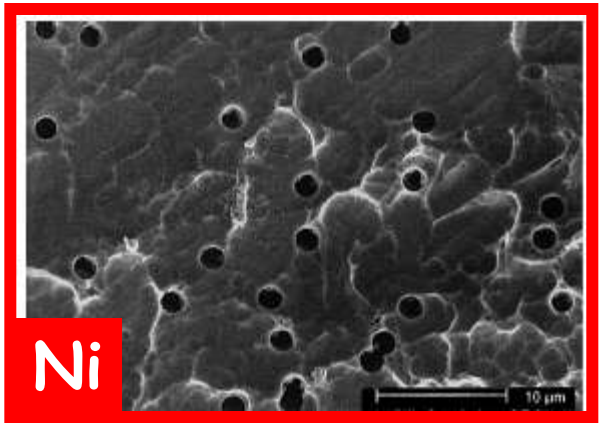
Collaborators:

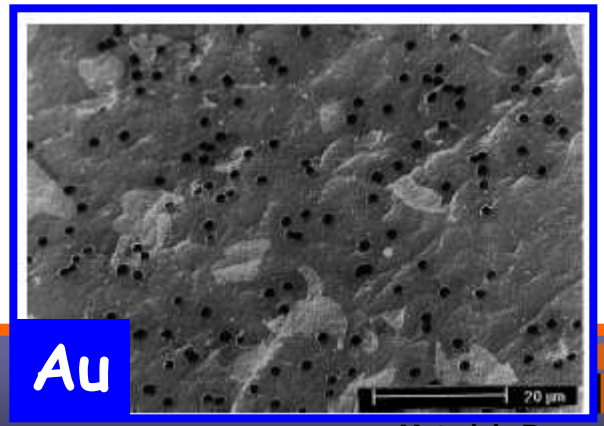
Prof. G. Mueller (Field Emission, Uni Wuppertal),
Prof. A.M. Pucci (IR spectroscopy, Uni Heidelberg),
Dr. W. Sigle (TEM, MPI, Stuttgart)
Prof. F. Voelklein (Thermoelectrics, Uni Wiesbaden)
Dr. Doug L. Medlin (TEM, Sandia National Labs)

Metal membranes by double replication









J. Vetter, D. Dobrev, NIMB 156 (1999) 177

Materials Research

Synthesis and Characterisation of Low Density Porous Polymers Made by Reversible Addition Fragmentation Chain Transfer (RAFT)

Wigen Nazarov[1], Christopher Musgrave[1], Kimberley L. Anderson[2], Nick Bazin[3], Douglas Faith[3]

[1] *University of St Andrews, Unit 4, High Energy Laser Materials Laboratory, North Haugh, St Andrews, Fife, KY16 9ST, UK.*

[2] *Department of Pure & Applied Chemistry, University of Strathclyde Thomas Graham Building, 295 Cathedral Street, Glasgow, G1 1XL, UK.*

[3] *Atomic Weapons Establishment, Reading, RG7 6DP, UK.*

Aims and objectives

- To synthesise a polymeric foam with high degree of polydispersity index using RAFT reagent.

$$\text{PDI} = \frac{\overline{M_w}}{\overline{M_n}} \geq 1$$

- To use RAFT synthetic method and investigate the effect of RAFT reagents for the production of polymeric foams, specifically poly-HIPE foams
- To investigate the differences between HIPE foam synthesised using RAFT reagents and HIPE foams synthesised by free radical polymerisation – FRP

Foams made from High Internal Phase Emulsions HIPE

General:

- HIPEs have high strength at low density in comparison to other foams
- They have an open pore cellular structure and have been used in laser targets for many years
- All PolyHIPE used in laser targets have been synthesised from Styrene, Chlorostyrene and Divinyl Benzene monomers,

Styrene Methyl methacrylate polyHIPE investigated in this Project:

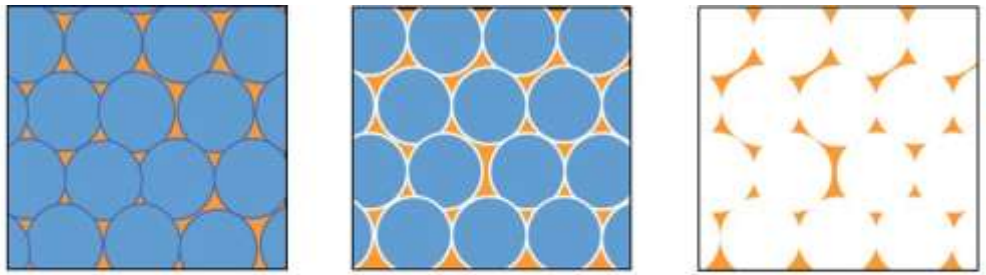
- Reasons for using Styrene Methyl Methacrylate co-polymers:
  1) It is a well characterised co-polymer system,
  2) Because measurements of polydispersity was important, Styrene-co-methyl methacrylate is soluble in many solvents, but styrene-co-DVB not as soluble

High Internal Phase Emulsions HIPE

HIPE is an emulsion which consists of a dispersed (internal) and continuous phase stabilised by an appropriate surfactant.

For an emulsion system to qualify as a HIPE the dispersed (internal) phase must occupy more than 75% by volume. Typically most HIPEs are about 90% internal phase.

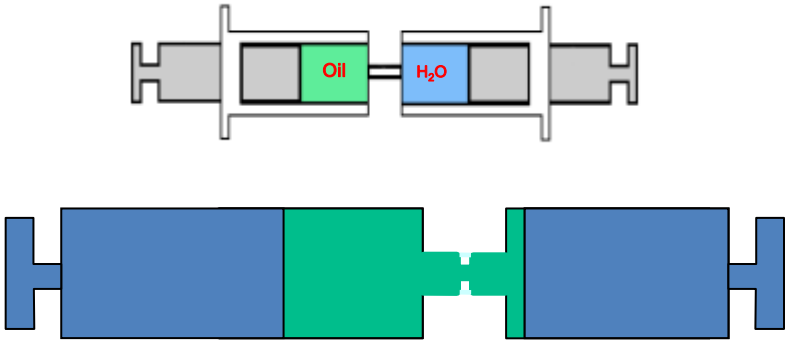
In the production of foams using HIPEs the internal phase is the pore former and the continuous phase is the polymeriseable monomer (or monomers), stabilised by a surfactant and an initiator.



Emulsification

1. Syringe Method

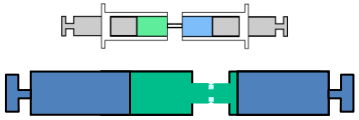
The emulsion is produced by the shear force created when the liquids squeeze through the small orifice of the gas tight syringes, which are connected by a luer lock.



2. Mechanical Stirrer Method

The emulsion is produced by slow addition of the aqueous phase to the continuous oil phase, whilst stirring at moderate shear.

Steps in Foam Production



emulsify

Place in airtight containers and Cure at 60 °C

Soxhlet Solvent extraction

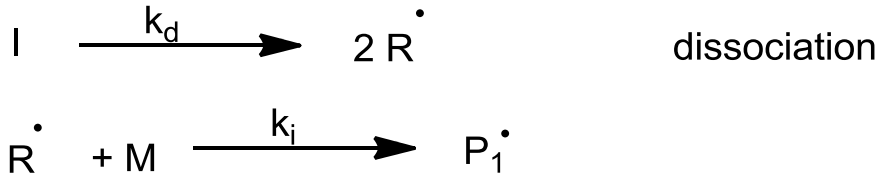
Dry in moderate Oven or air

Difference between FRP and RAFT polymerisation

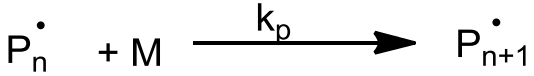
Free Radical Polymerisation

ALL polyHIPE Foams made for Targets are made by Free Radical Polymerisation FRP

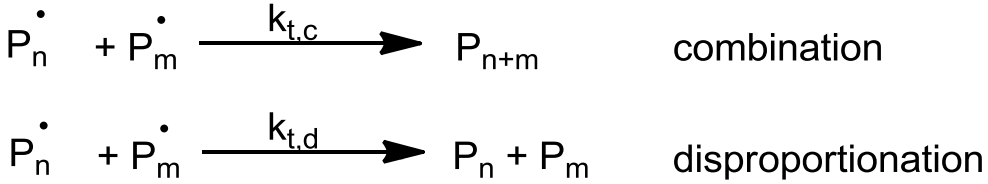
Initiation



Propagation

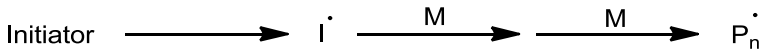


Termination

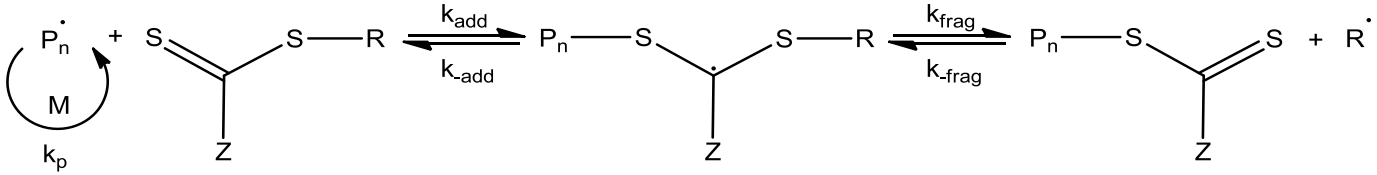


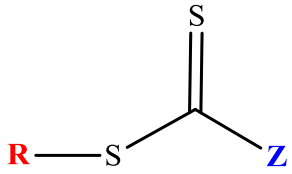
Controlled Radical Polymerisation and the RAFT process

Initiation



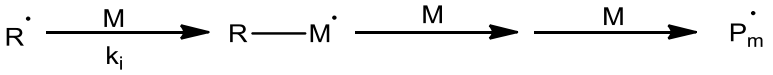
Reversible chain transfer/propagation



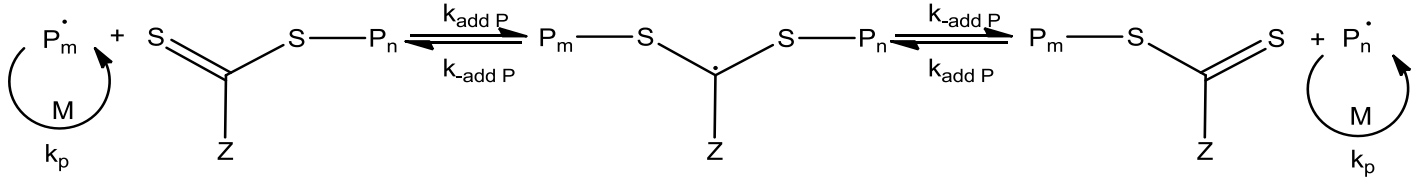


General Structure of RAFT reagent, **free radical leaving group R** and a **group Z which modifies the reactivity** of the C=S bond

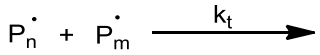
Reinitiation



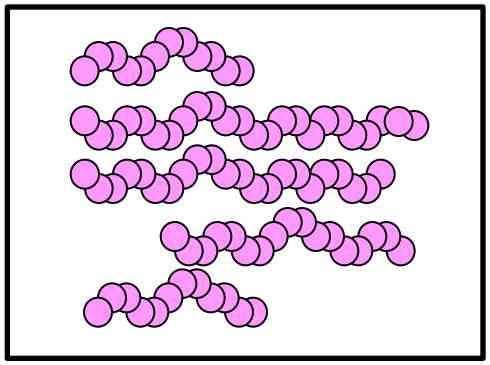
Chain equilibrium/propagation

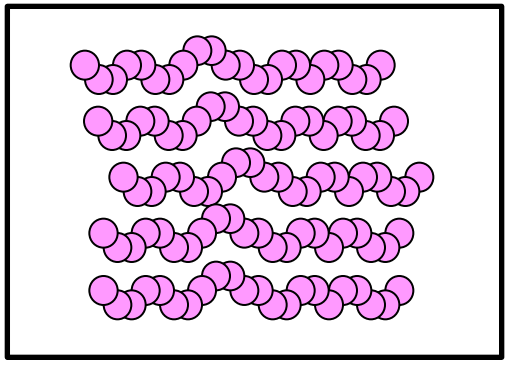


Termination



Comparisons between FRP and RAFT





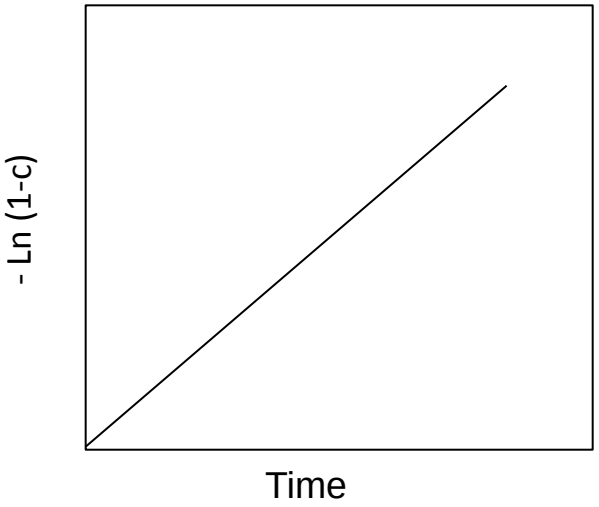
Free radical polymerisation

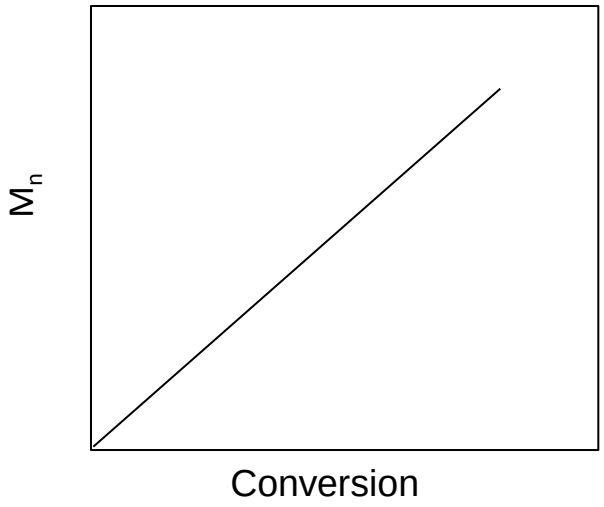
- Not a living process
- Cannot form architectured polymers such as block copolymers
- Broad molecular weight distribution, high polydispersity index

Controlled radical polymerisation

- Living process
- Able to produce architectured polymers such as block copolymers
- Controlled molecular weights obtained, low polydispersity index

Effect of RAFT reagent - Controlled polymerisation





- A plot of –ln(1-conversion) against time should show linear relationship
- The molecular weight (M_n) should evolve linearly with conversion, close to theoretical prediction

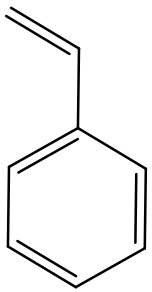
This trend is observed because the rate of polymer growth is constant and not dependant on monomer concentration but on

$$\frac{[\text{Monomer}]}{[\text{RAFT agent}]}$$

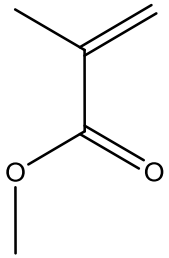
- Polydispersity index should decrease towards 1 as conversion increases, with final value <1.5

Polymer system prepared by RAFT polymerisation

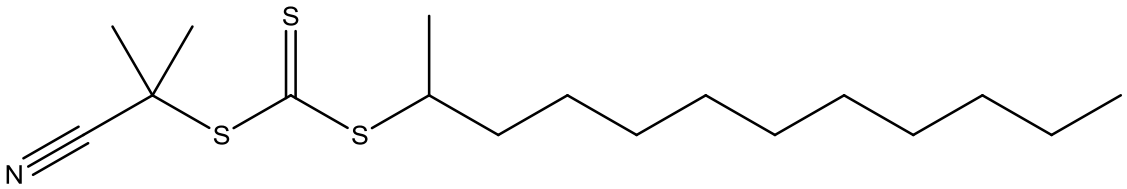
Monomers	**Molar Ratio of Monomers**	**Polymer**
styrene + methyl methacrylate	1:1, 2:1	PS-*co*-PMMA
styrene + methyl methacrylate (by RAFT polymerisation)	2:1	PS-*co*-PMMA



Styrene



Methyl Methacrylate (MMA)



S-(2-cyano-2-propyl)-S-dodecyltrithiocarbonate

Choosing the reaction conditions for RAFT Polymerisation concentrations used

The ratio of RAFT agent to initiator must be kept high, to minimise the free radical interactions which results in termination

The ratio of [monomer] to [RAFT agent] pre determines the maximum number average molecular weight (M_n) which will be reached.

$$Targettted\ \ degree\ of\ Polymerisation = \frac{[monomer]}{[RAFT\ agent]}$$

$$M_{n\ (calc)} = \left(degree\ of\ polymerisation\ x\ M_{(monomer)}\right) + M_{(RAFT\ agent)}$$

For polymerisation of styrene and Methyl Methacrylate using RAFT reagents the following different concentrations of RAFT reagents used:

Predicted $M_{n\ calc}$ = 30 365 g/mol - Small
Predicted $M_{n\ calc}$ = 141 119 g/mol - Medium
Predicted $M_{n\ calc}$ = 207 213 g/mol - Large

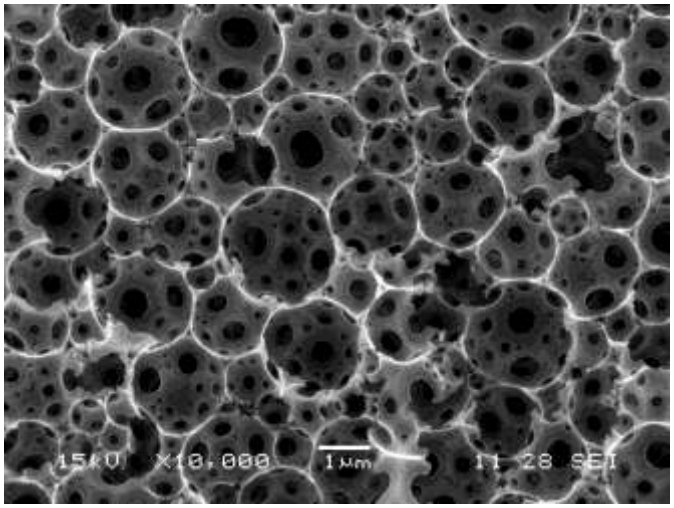
Results and characterisation

Results – GPC

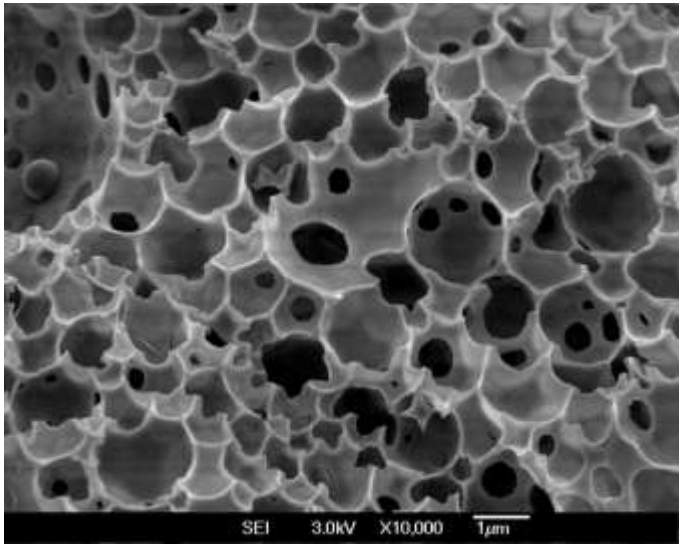
Sample	Molecular Weight Distribution (Polystyrene Equivalent) / Daltons				
	M_n	M_w	M_z	M_p	PDI
PS-*co*-PMMA FRP	113 900	533 000	1 892 300	178 700	4.68
PS-*co*-PMMA RAFT Small Peak 1	18 700	27 300	36 100	25 900	1.46
PS-*co*-PMMA RAFT Small Peak 2	Insufficient peak resolution			570 400	
PS-*co*-PMMA RAFT Medium Peak 1	81 150	159 230	268 760	130 000	1.96
PS-*co*-PMMA RAFT Medium Peak 2	Insufficient peak resolution				
PS-*co*-PMMA RAFT Large Peak 1	44 000	91 300	152 700	80 600	2.08
PS-co-PMMA RAFT Large Peak 2	Insufficient peak resolution				

Results - SEM images

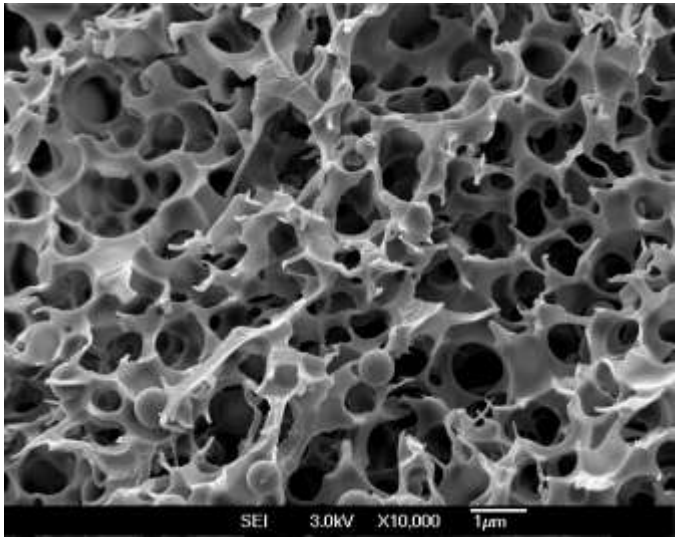
In all micrographs, Magnification = 10,000 Marker = 1 µm



PS-*co*-DVB HIPE



PS-*co*-PMMA RAFT Small



PS-*co*-PMMA HIPE Normal

TDNMR results for Styrene-MMA polyHIPE foam

Material	Polymerisation method	T_1 relaxation time (ms)	$T_{1\rho}$ relaxation time (ms)	T_2 relaxation time (ms)
2:1 S:MMA	FRP	449 ± 3	0.7 ± 0.2	$0.0083 \pm 8 \times 10^{-5}$
			6.6 ± 0.2	
	RAFT small	320 ± 9	0.7 ± 0.2	$0.0099 \pm 2 \times 10^{-5}$
			6.3 ± 0.3	
	RAFT medium	440 ± 10	0.9 ± 0.3	$0.0081 \pm 1 \times 10^{-5}$
			7.0 ± 0.4	
	RAFT large	458 ± 10	0.5 ± 0.1	$0.0082 \pm 1 \times 10^{-4}$
			6.4 ± 0.2	

Summery and Future Work

- It has been demonstrated that the Production of polyHIPEs using RAFT Reagents is possible and foams with low PDI are produced
- **<u>Improvements and refinements</u>**
  - ❑ Span 80 was used as a surfactant for the production of SMMA polyHIPE, However, SEM of SMMA PolyHIPE shows that there is some indication of emulsion breaking before polymerisation completed. Other surfactant, such as block co-polymers can be used which might considerably improve the polyHIPE structure of RAFT synthesised SMMA.
  - ❑ Using different RAFT concentrations to find the optimum PDI
  - ❑ Measurements of the strength of SMMA polyHIPE made using RAFT reagent and compare with SMMA polyHIPEs made by FRP to see whether the structural strength of SMMA polyHIPE has improved using RAFT reagents

Acknowledgment

- Most of the synthetic work on RAFT was carried out by Kimberley Anderson and TDNMR work by Christopher Musgrave.
- We would like to thank all AWE characterisation team for carrying out GPC and SEM
- Special thanks to AWE for continuous financial support for High Energy Laser Materials Laboratory at St Andrews