JOANNEUM RESEARCH

Laser Center Leoben

Jožef Stefan Institute

Alpbach Technology Forum 2007

Workshop: "Technology Transfer in the European Region Adria-Alpe-Pannonia – Challenges, Opportunities and Initiatives"

Cooperation between JOANNEUM RESEARCH and Jožef Stefan Institute in Deposition and Characterisation of Thin Films

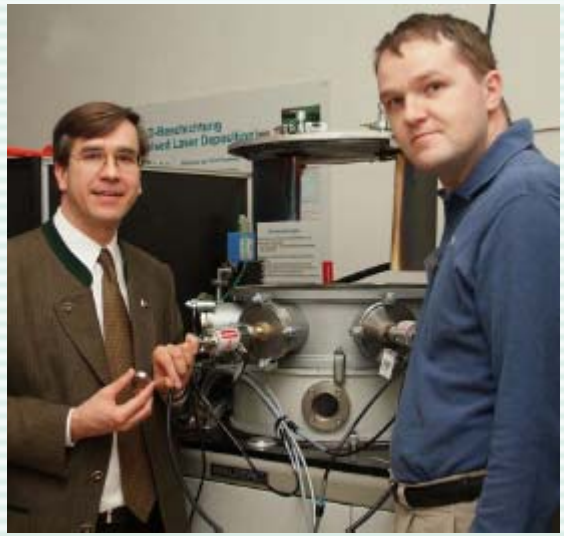
Wolfgang Waldhauser (JOANNEUM RESEARCH)
Miha Čekada (Jožef Stefan Institute)

ISO 9001 certified

a TRADITION of INNOVATION

Outline

- **Short Introduction**
  - Laser Center Leoben (JOANNEUM RESEARCH (JR))
  - Department of Thin Films and Surfaces (Jožef Stefan Institute (IJS))
- **Project Presentation**
  - Motivation and aims of the project
  - Benefits for JR and IJS
  - Fundamentals of Anode Layer Source
  - Results
  - Future joint projects



- **Premises for a successful cooperation in R & D**
- **Premises for a successful technology transfer**

Laser Center Leoben

JOANNEUM RESEARCH Forschungsgesellschaft mbH

Company (as of 30/6/2006)

- Owners: Province of Styria (90%), TNO (10%)
- Institutes: 14 Staff: 384
- Revenues: 31,3 Mio EUR
- Projects per year: approx. 380
- Publications per year: approx. 200

Divisions

DIV1: Sustainability and Environment
DIV2: Information Technology
DIV3: Electronics and Sensor Technology
DIV4: Materials and Processing
DIV5: Economy and Technology
DIV6: Medical Technology

Das Land Steiermark

Archduke John (1782 – 1859)

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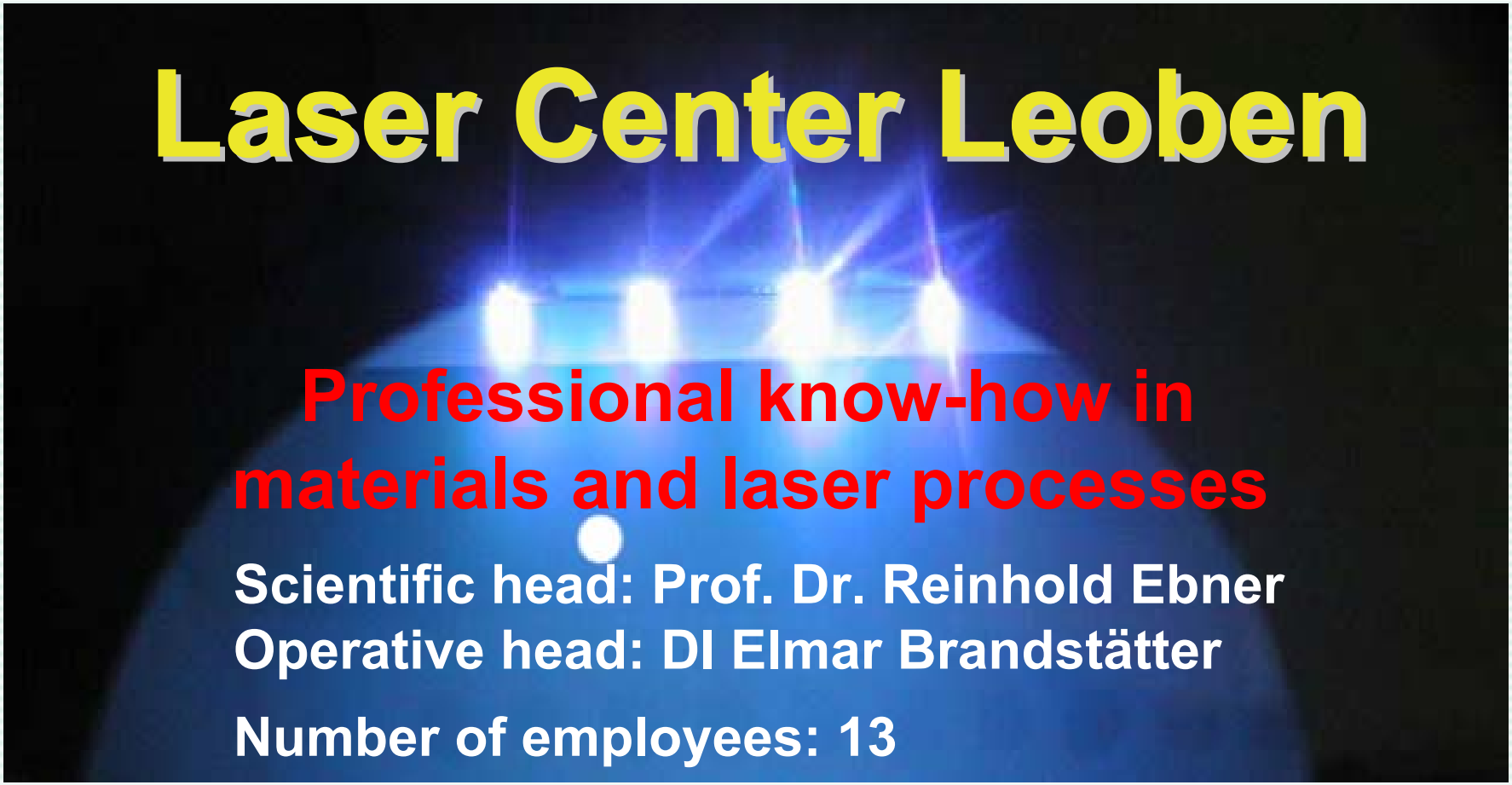
Presentation of the Institute
www.joanneum.at/LZL



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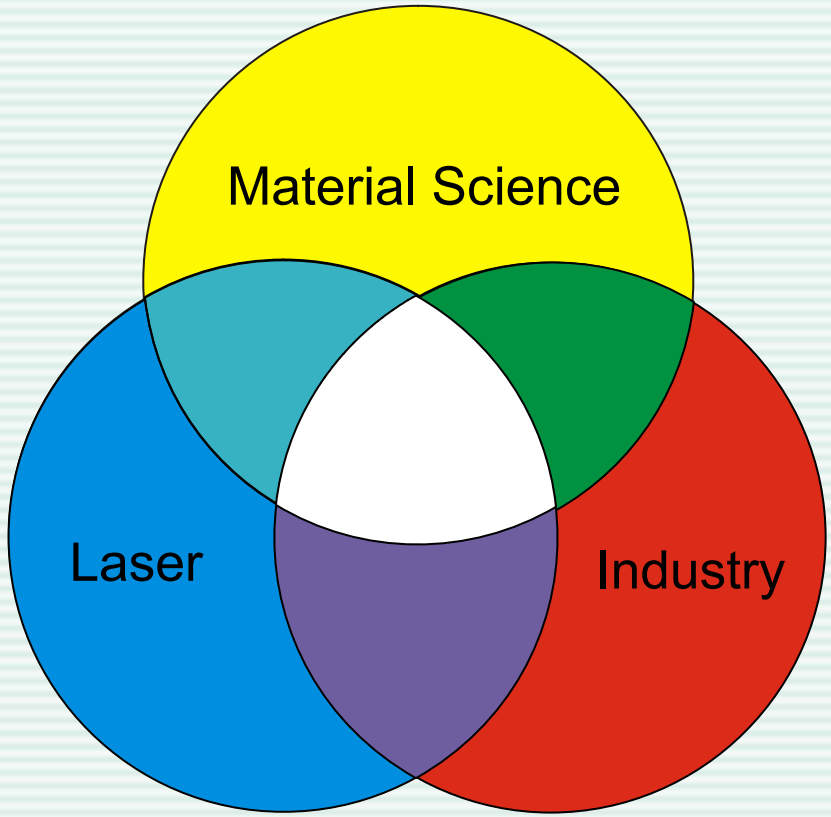
Laser Center Leoben

- Founded in 1989 in cooperation with the University of Leoben
- Industrial laser applications
  - Laser Welding
  - Laser Cladding
  - Laser Rapid Tooling
  - Laser Thin Film Technology

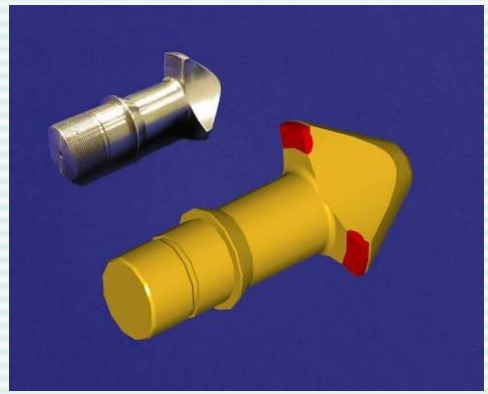


Main Areas of Research

Laser Materials Processing

→ 2D/3D laser production technology

- laser processing (welding, cladding, alloying, rapid tooling)
- process development and optimisation
- design of materials
- quality assurance in laser welding



→ **Laser thin film technology** (Pulsed Laser Deposition)

- thin film deposition, low temperature coating
- process development and optimisation
- development of functional thin films
- film characterisation

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Laser Thin Film Technology

Scientific and technological goals:

Development:

- Functional thin films (anorganic, organic)
- **Room-temperature deposition techniques** (Pulsed Laser Deposition (PLD))
- Hybrid PVD techniques (e.g. PLD + Sputtering)

Biocompatible coatings

Transfer to industrial applications:

Coating of forming tools

- Tribological, wear-resistant and functional coatings on polymer substrates
- Functional coatings for medical and biological applications
- Sensor coatings
- Laser and plasma-assisted coating processes

Coated tools

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Laser Thin Film Technology

Equipment for Film Deposition

- Laboratory PLD coating plant (PLD, DC Sputtering, RF Sputtering, Diameter of sputtering cathodes 76 mm)
- Demonstration PLD coating plant (PLD employing 4 laser beams, rectangular sputtering cathode 73.6 mm x 431,8 mm, recipient diameter 740 mm, coating height 300 mm)
- Q-switched laser systems
  - 1000 mJ Nd:YAG laser
  - 1200 mJ Nd:YAG laser
- **Projected** (nSEC Leoben)
  - Multi-functional PLD coating plant
  - Excimer laser (248 nm)

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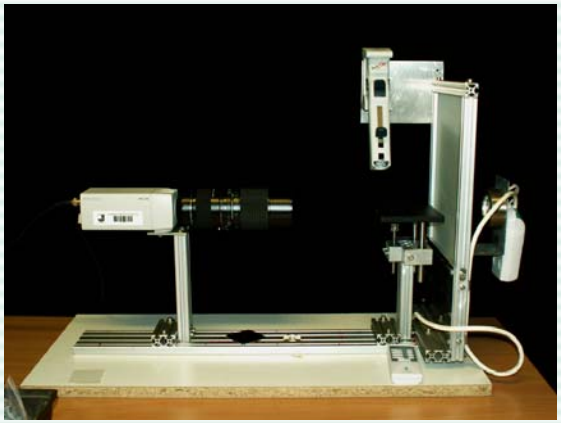
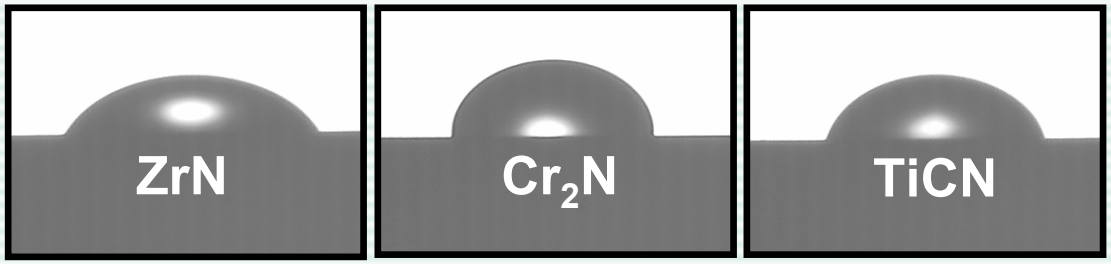
Laser Thin Film Technology

Equipment for Film Characterisation

- Scratch test
- Pin-on-Disc-test, linear tribometer
- Spherical calotte grinding equipment for film thickness measurement
- Equipment for Contact angle measurement
- Colorimeter



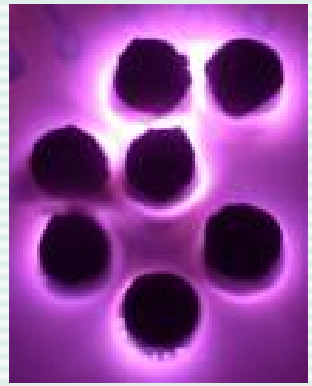
Contact angles (deionized water)



Presentation of the Jožef Stefan Institute

Jožef Stefan Institute

- the biggest R&D institute in Slovenia
- research fields:
  - physics
  - new materials and nanotechnology
  - chemistry and biochemistry
  - environmental sciences
  - electronics and information technologies
  - reactor engineering and energetics



Jožef Stefan (1835–1893)

- Stefan-Boltzmann law
- heat conductivity of gases

Presentation of the Jožef Stefan Institute

Basic facts

- 800 employees, ~1/2 PhDs
- budget 38 M€, 71% ministry, 16% industry, 13% international

Achievements

- 1200 papers and 30 patents per year
- so far 2000 BSc and 700 PhD theses (30 per year)

International cooperation

- 190 multilateral projects (6FP: 89)
- 170 bilateral projects (HR: 23, USA 18, F 15, A 8)

Co-founder of the Jožef Stefan postgraduate school

Jožef Stefan Institute

Presentation of the Department of Thin Films and Surfaces

- **Research areas**
  - ➔ hard protective coatings for tools
  - ➔ QC thin films, SOFCs, diffusion, corrosion, plasma diagnostics
- **Hard coatings center**
  - ➔ serving our industrial partners in protection of tools
- **Staff**
  - ➔ 4 scientists, 2 PhD students, 4 technicians
  - ➔ head: Dr. Peter Panjan
- **Budget**
  - ➔ 55 % industry, 40 % ministry, 5 % EU

Deposition techniques

thermoionic arc
ion plating
Balzers BAI 730

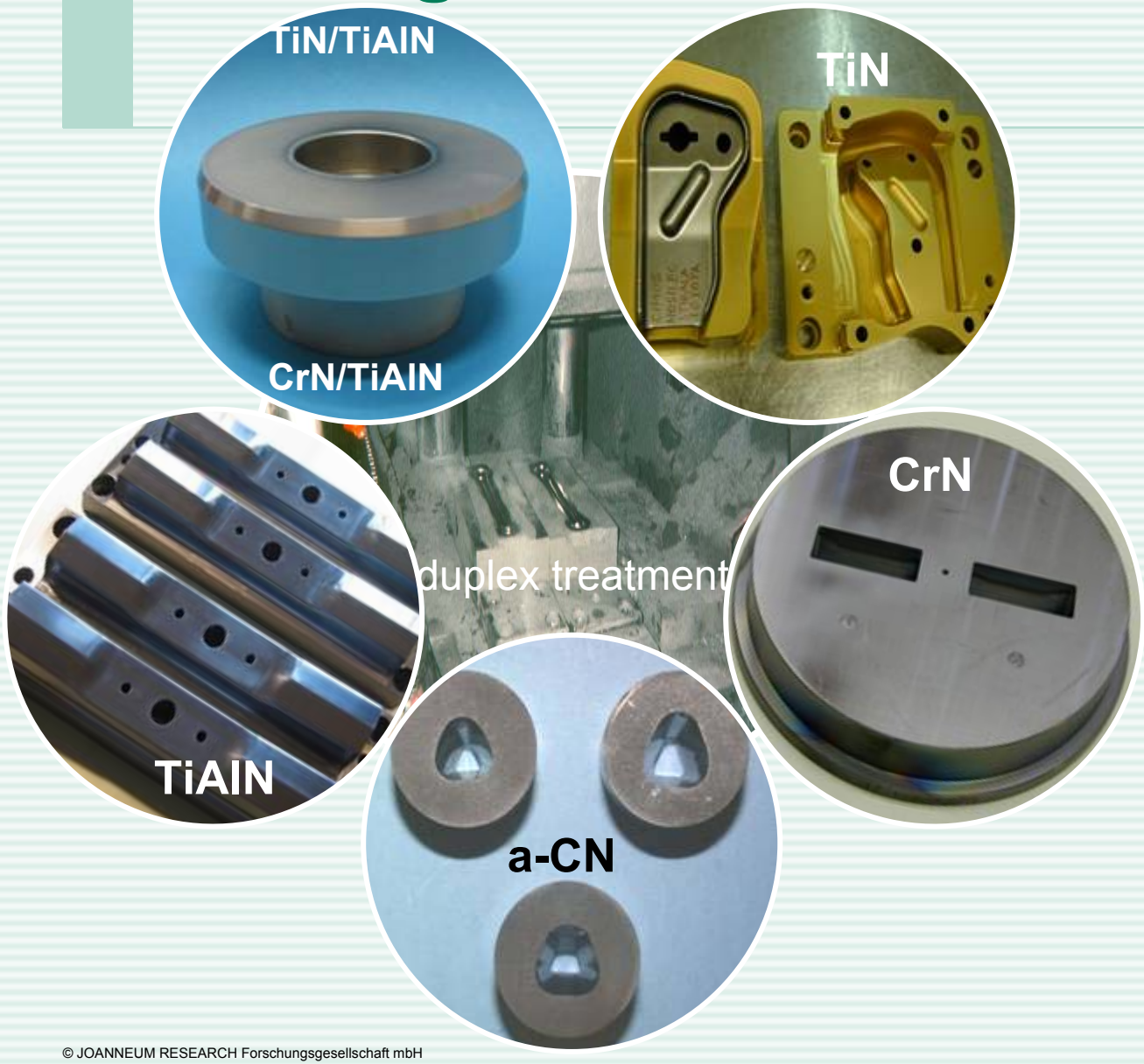
plasma cleaning
in BAI 730

magnetron and
e-gun
Balzers BAK 600

unbalanced
magnetron
sputering
CemeCon CC 800

triode sputtering
Balzers Sputron

Coatings offered

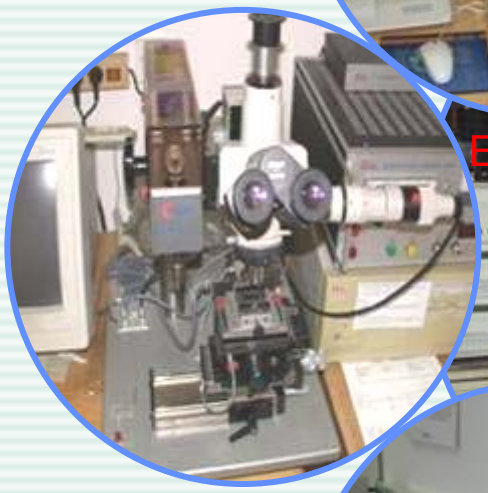


Characterisation equipment

Nanohardness tester

3D stylus profilometer

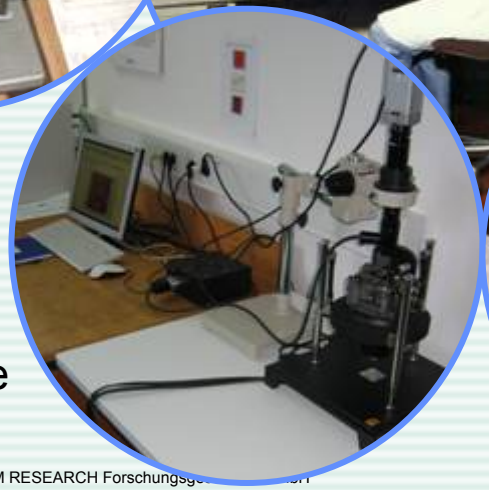
Adhesion tester



Energy and mass spectrometer

conventional hardness tester

AFM microscope



Rockwell hardness tester

Project description

Grant programme within the brainpower Austria initiative

- **Titel:** Deposition and characterisation of functional carbon based coatings
- **Period:** January 2007 – March 2007
- **Project costs:** € 11.752,-
- **Motivation of the project:**
  - ➔ The Thin Film Group of LZL is working in the field of the development and characterisation of functional coatings
  - ➔ Carbon based coatings (DLC = Diamond-like carbon) are a main research field
  - ➔ Pulsed Laser Deposition and Magnetron Sputtering are well established coating techniques at LZL

Project description

- An ion gun based on the principle of the Anode Layer Source (ALS) is available at LZL and has been used for substrate pre-treatment (ion etching)
- There was no experience in employing this equipment for direct film deposition at LZL and IJS
- IJS has no competent experience in DLC coatings

Scientific aims of the project:

- Deposition of DLC coatings by employing ALS
- Characterisation of the coatings
- Comparison of this technique with other coating techniques

Project description

General aims of the project:

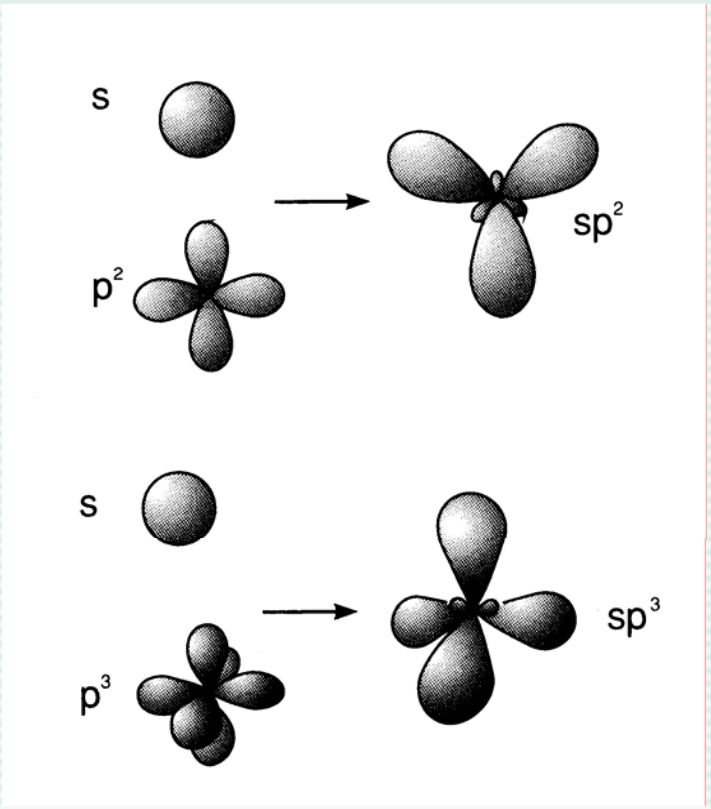
- Building up of a long-term collaboration with Jožef Stefan Institute
- Definition and application of future joint projects

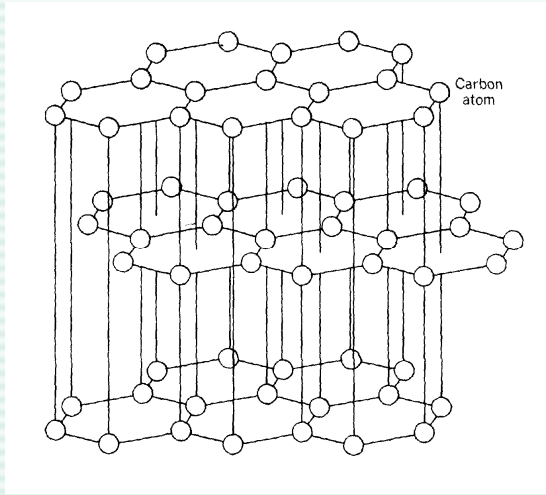
Benefits for LZL and IJS

- Access to film characterisation methods (XPS, neutron activation, profilometry) (LZL)
- Extended knowledge on carbon based coatings (IJS)
- Comparison of different coating techniques (both)
- Exchange of experience and knowledge (both)
- New collaboration (both)
- Possibility for future joint projects (both)

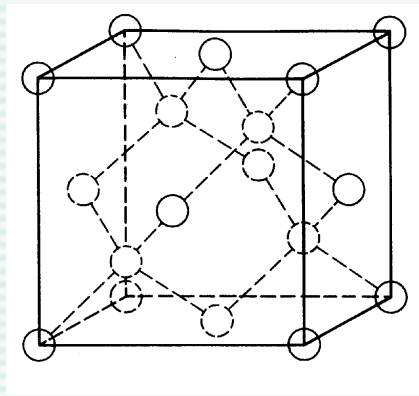
Chemical bonds of carbon

Hybridisation





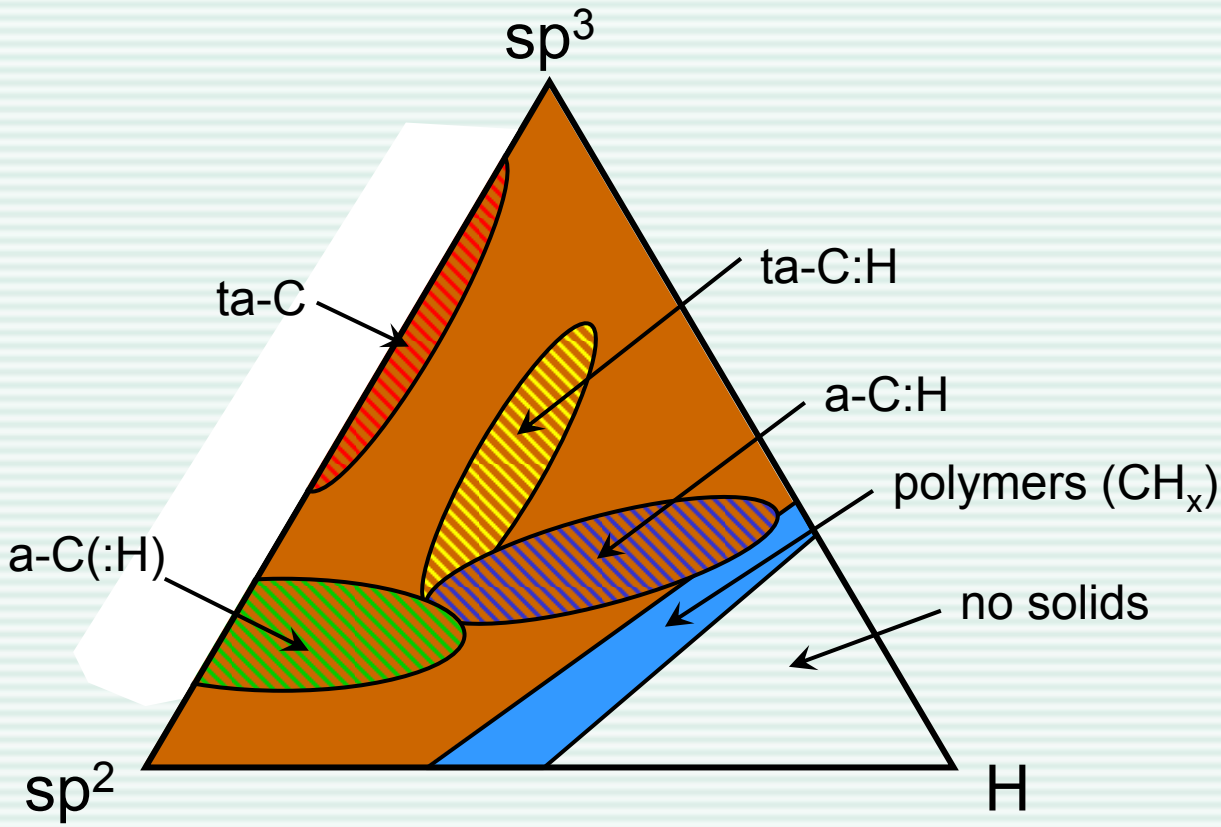
Graphite



Diamond

Diamond-like Carbon (DLC)

Ternary phase diagram of amorphous C-H alloys



graphite, soot,
fulerenes, nanotubes

Typical properties of DLC

Density	1,8 - 3,5 g/cm³
Hardness	5 - 80 GPa
Elastic modulus	140 - 600 GPa
Friction coefficient	0,01 - 0,2
Reactivi	
Permeal	
Thermal	
Thermal expansion coefficient	$9 \cdot 10^{-6}$ °C
Electrical resistance	10^{2} - 10^{16} Ωcm^{-1}
Dielectric coefficient	< 4
optical Transparence	Vis and IR; optical bandwidth 1,0 - 4,0 eV
Refraction index	1,8 - 2,3
Biocompatibility	total cell integration, no defense reactions

Tailoring of these properties by varying sp^2 / sp^3 carbon bonding ratio

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FORSCHUNG AUSTRIA
Zukunft verbindet.

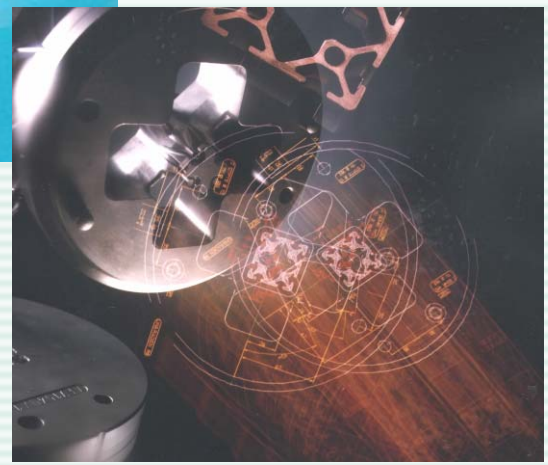
Jožef Stefan Institute

Potential applications of DLC coatings

Drills

Medical Implants

Roller and journal bearings



Forming tools

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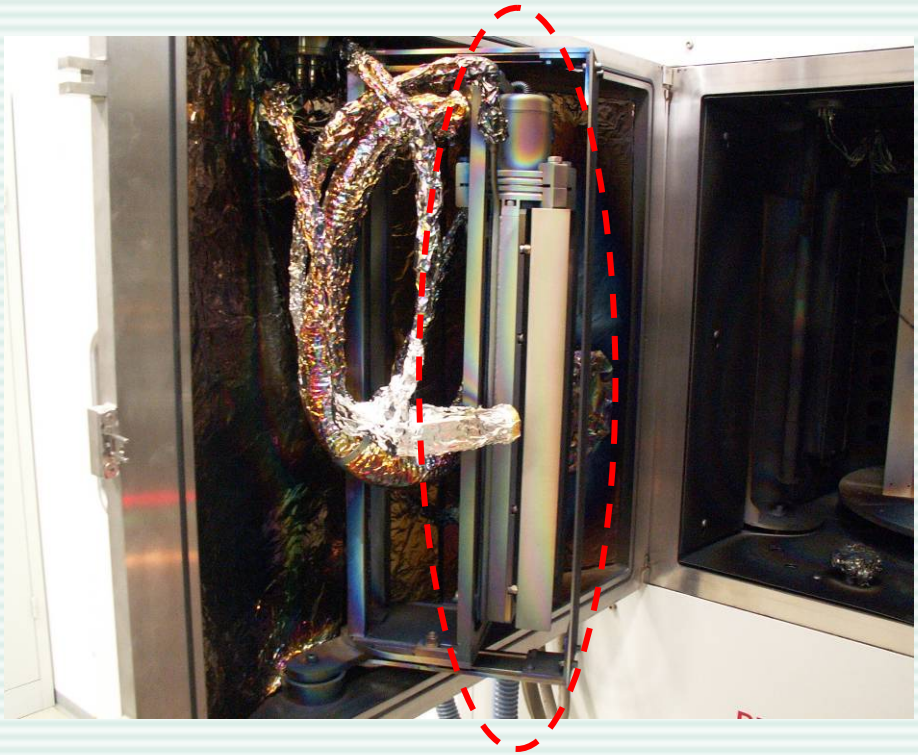
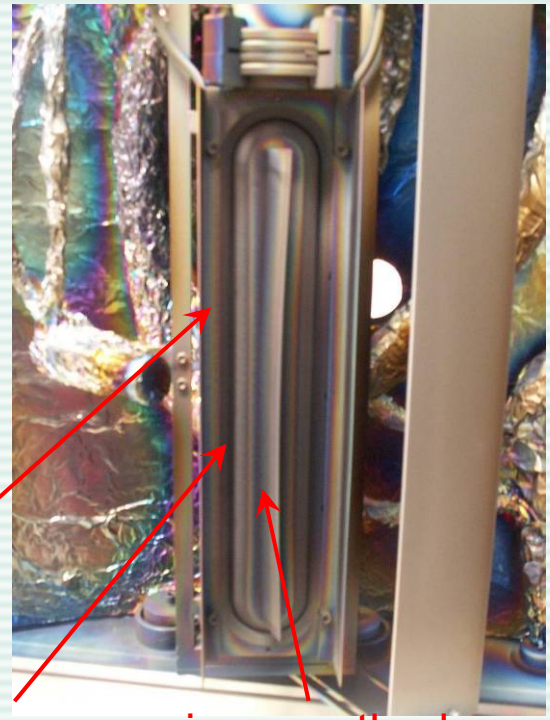
Background of the experimental technique

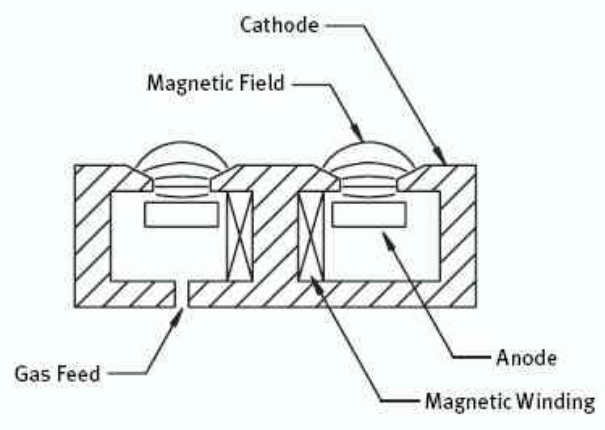
- **Idea in the 1960's: to construct an ion propulsion system for space satellites**
  - ➔ Kaufmann ion source (USA) : Anode layer ion source (USSR)



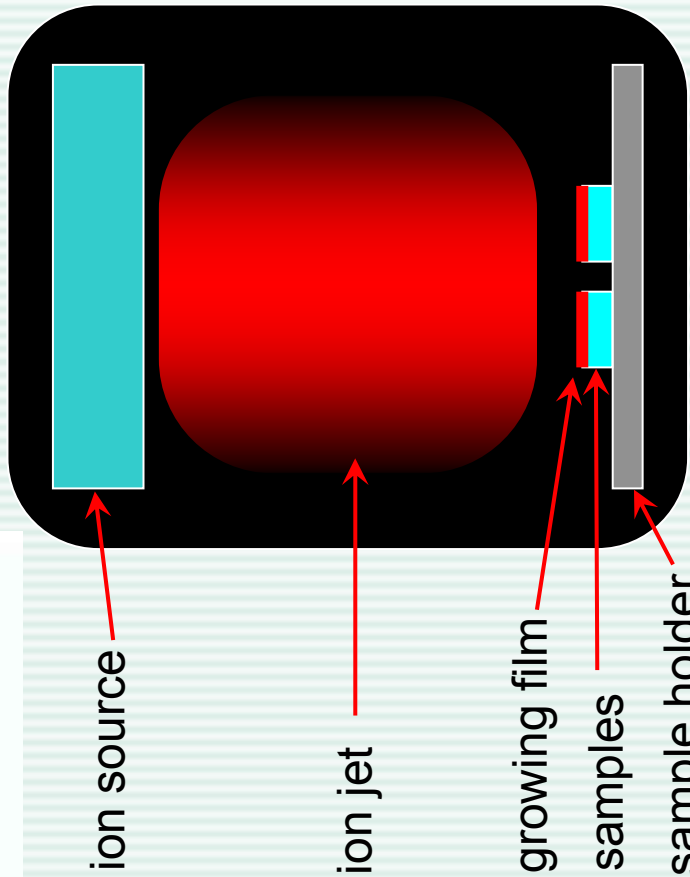


Background of the experimental technique

Cross-section of the anode layer source



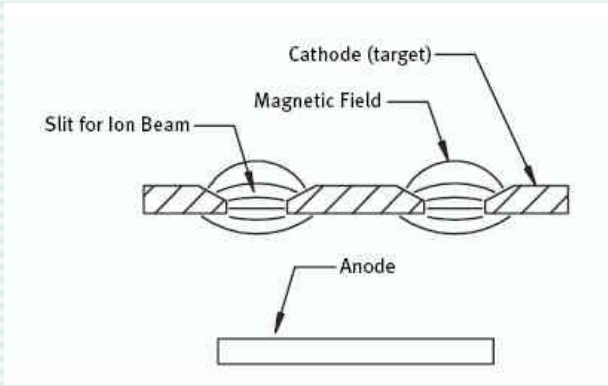
Geometry in the chamber



Expected benefits:
- easy, robust
- no target needed
- no droplets
- no contaminants
- good adhesion

Possible drawbacks:
- unexplored area
- large internal stress

Detail of the slit



Film deposition by ALS

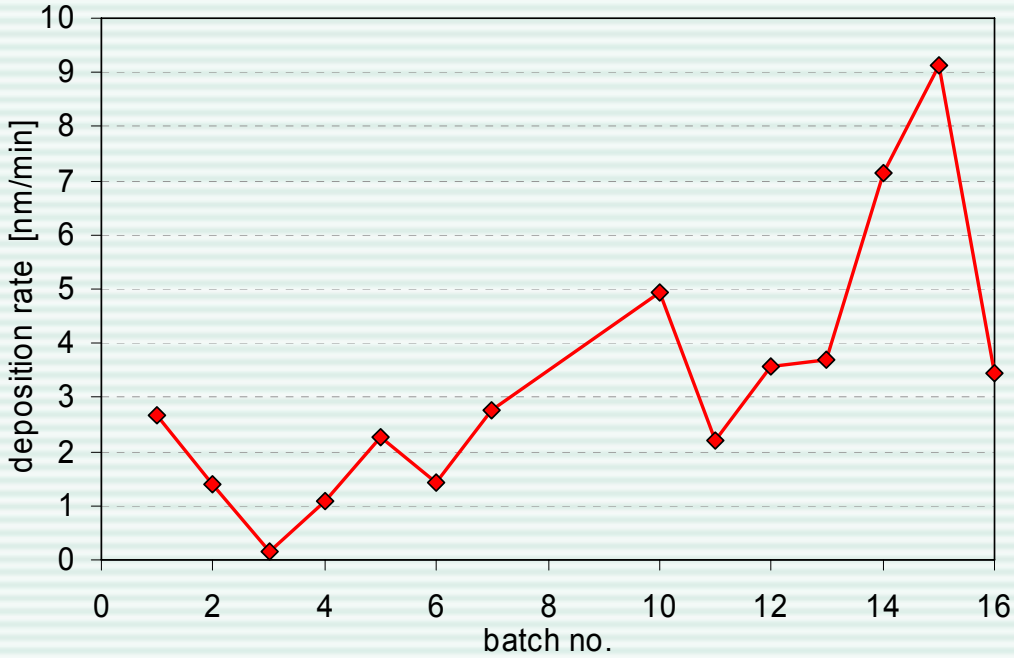
Parameters to be varied

- where to feed the C_2H_2 gas
- ratio Ar : C_2H_2 (or none Ar at all?)
- total flow of the gases
- cathode voltage
- sample movement:
  rotation, oscilation, static
- deposition time

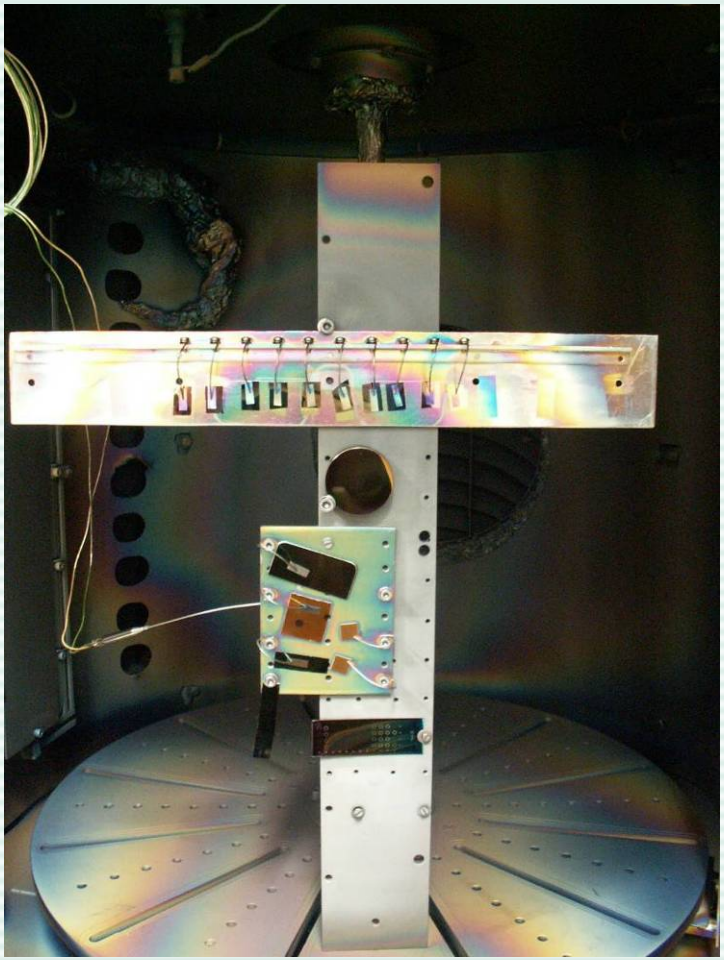
- constant parameters /
  two sets /
  periodic changing

What do we want to achieve

- a reasonable deposition rate
- acceptable adhesion
- other parameters for
  particular application

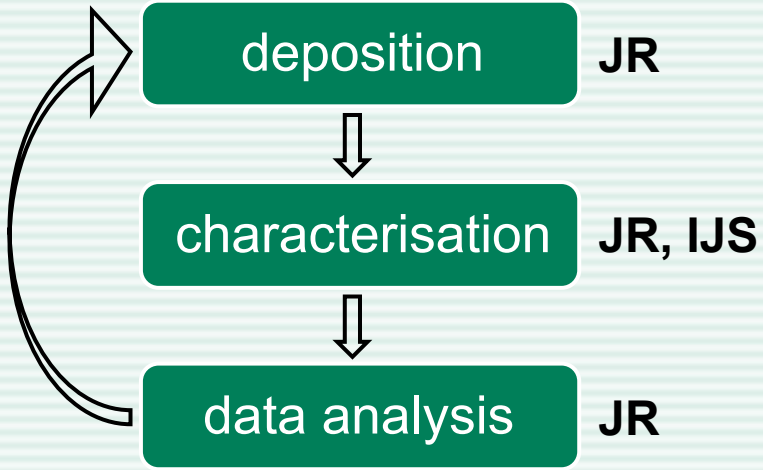


Film characterisation

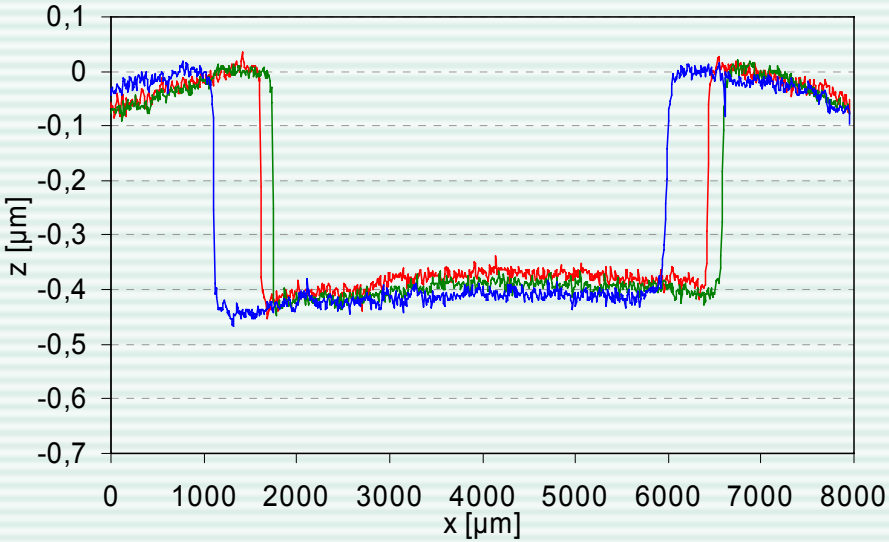


Properties to be measured

- Thickness
- Hardness
- Adhesion
- Internal stress
- Density
- Chemical bond (Raman spectroscopy)
- Friction and wear

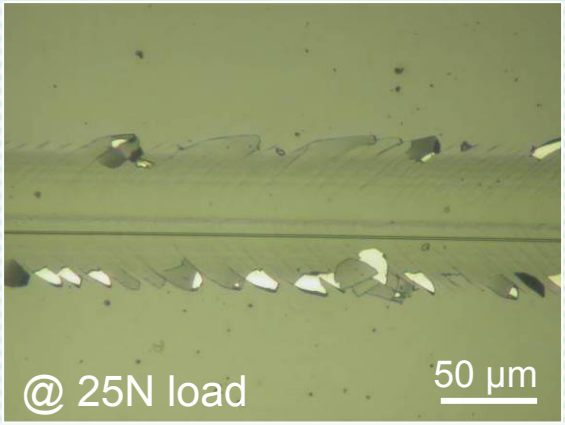


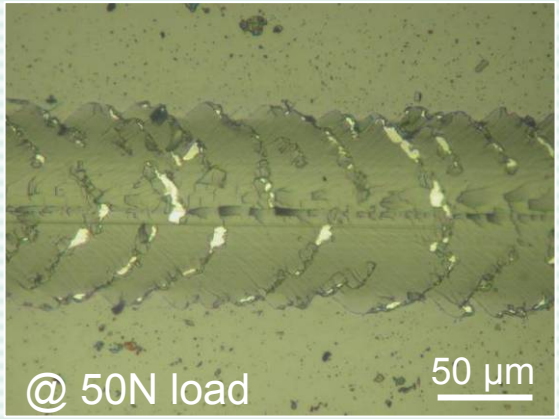
Film characterisation

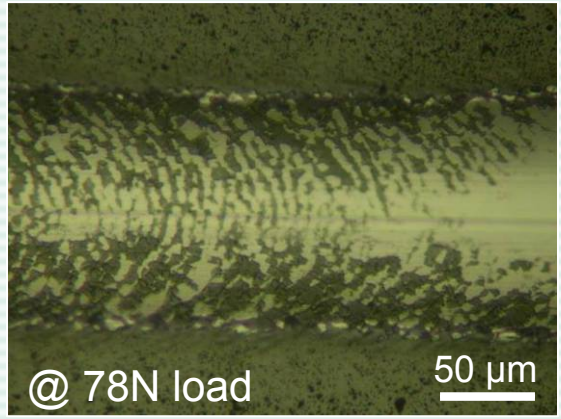


Thickness measurement

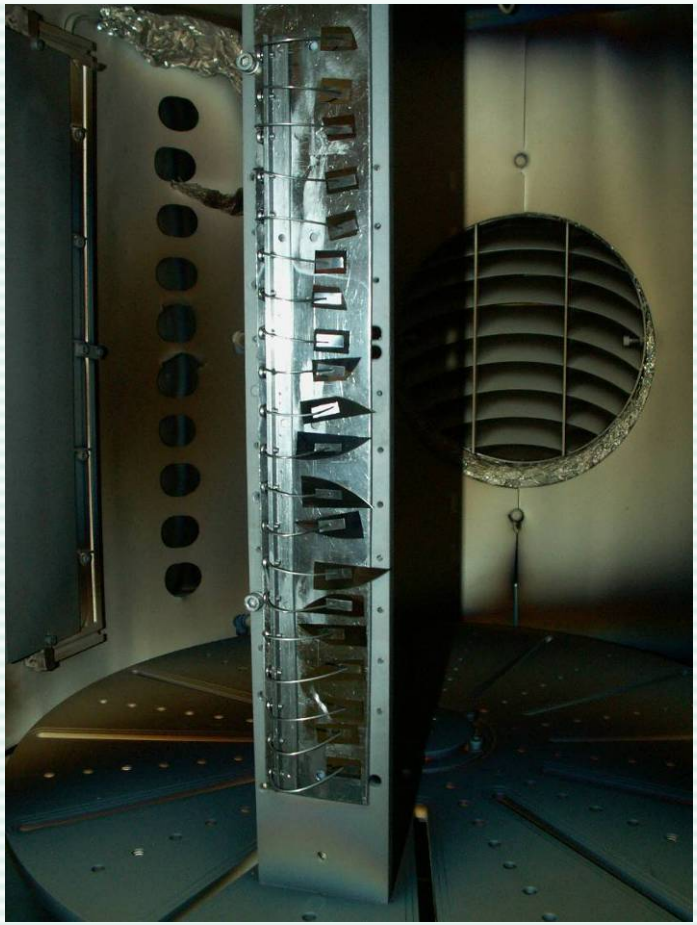
Adhesion measurement



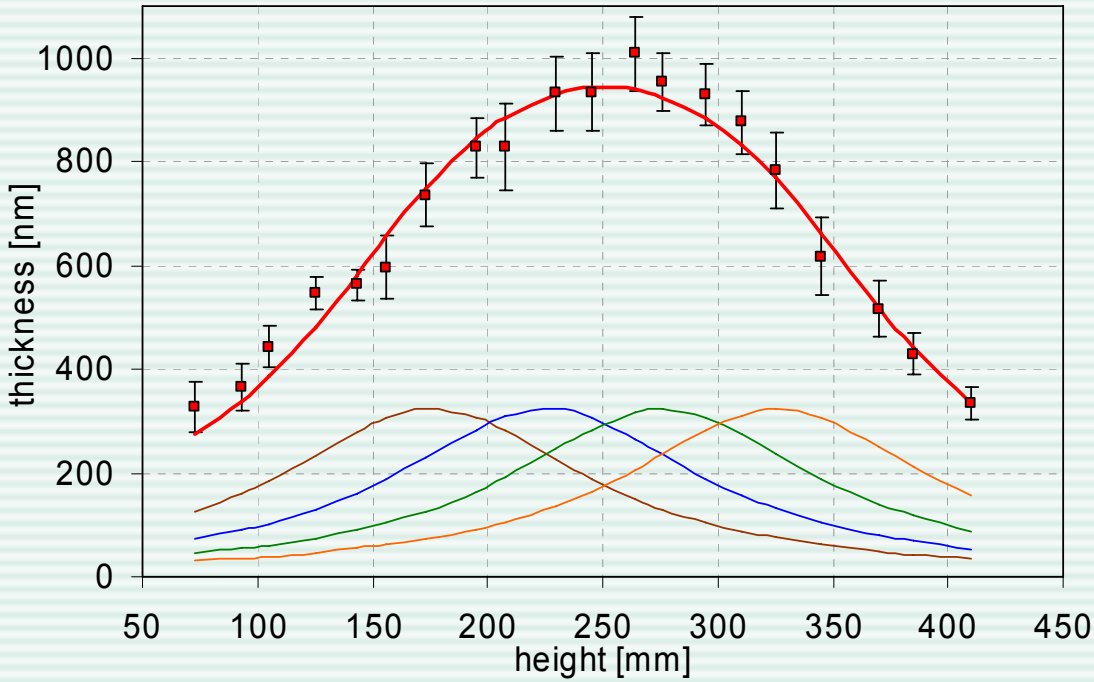




Other experiments



Profile uniformity of carbon coatings deposited by Pulsed Laser Deposition



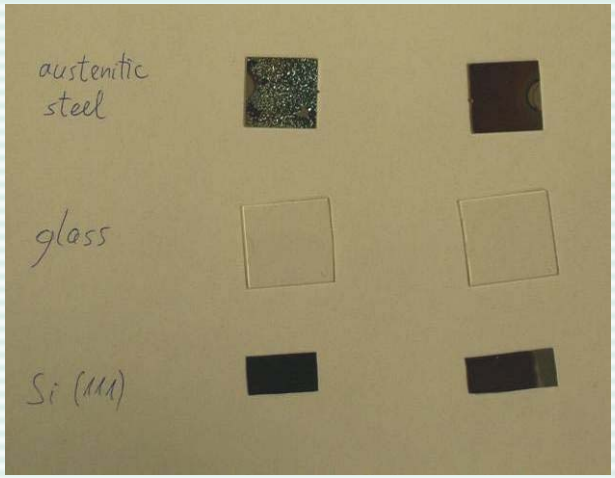
Conclusion and outlook

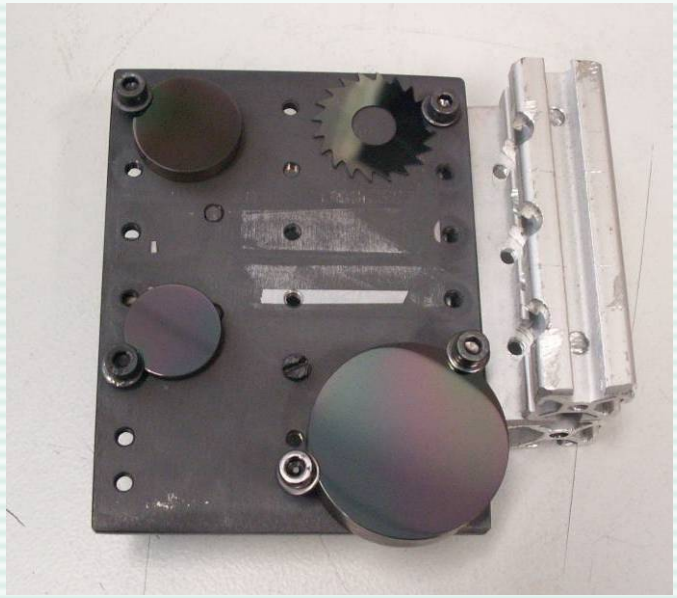
Work done so far

- Deposition of carbon coatings by ALS
- Established good collaboration on sample deposition and characterisation
- Definition of future joint projects

Work to be done

- Deposit coatings on real substrates
- Make additional characterisation not done so far
- Patent and/or publish





Future joint projects in preparation

MNT ERA Net Project

- **Acronym:** Hy-nano-IM
- **Title:** "Hydrogen-impermeable nano-material coatings"
- **Consortium:** Jožef Stefan Institute, JOANNEUM RESEARCH, Slovenian Steel company Acroni, Magna Steyr
  Status: submitted at recent call

COMET programme (Research Project)

- **Title:** "Improvement of hard coatings on highly stressed tools by employment of an Anode Layer Source"
- **Consortium:** Ionbond Austria, JOANNEUM RESEARCH, companies from Slovenia, Jožef Stefan Institute
- **Status:** planned project

Premises for a successful cooperation in R & D 1/2

- **Cooperativeness at all hierarchies in the involved organisations**
- **Well-defined benefits for all partners**
  - ➔ Transfer and exchange of knowledge
  - ➔ Access to new methods and technologies
  - ➔ Access to new markets
- **No direct competition between the partners**
  - ➔ Partners should have different core competences
  - ➔ Complementary activities and tasks
  - ➔ Complementary infrastructure for R & D
  - ➔ Partners should work along the value-added chain
  - ➔ Partners should be active in different markets

Premises for a successful cooperation in R & D 2/2

- **Well-defined project goals for all partners**
- **Well-defined project schedule and tasks**
- **Clear and strict regulations**
  - → Intellectual property rights
  - → Joint use of equipment
  - → Economic transfer of the project results
- **Personal relations between the researchers and project collaborators**
  - → Fairness, trust, sympathy
  - → Openness for foreign civilisations
  - → Willingness to travel

Technology transfer from research institutes to industry

Premises for a successful technology transfer

- Scientific excellence of the research institutes
- Detailed knowledge about problems and questions in industry
- Clear visions and imaginations about future demands of economy and society
- Availability of excellent technical equipment at the research institutes
- Clear and strict regulations
  - Intellectual property rights
  - Economic transfer of the project results
- Successful reference projects
- Clear and honest communication

Thank you for your attention!

Acknowledgements:

Financial support by

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- Slovenian Research Agency
- European Union

is highly acknowledged.