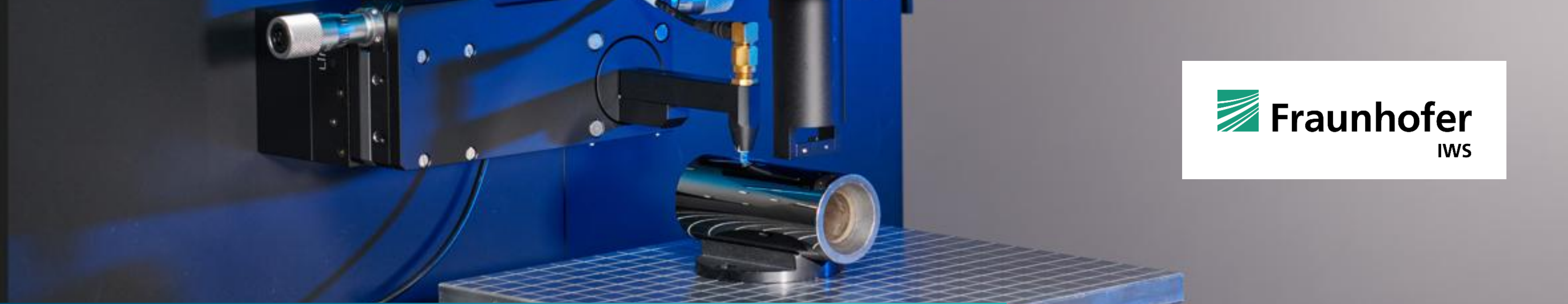
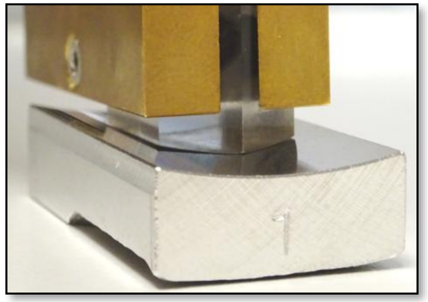
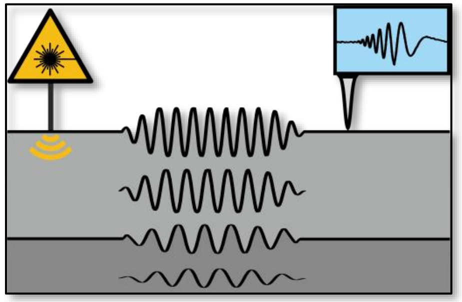


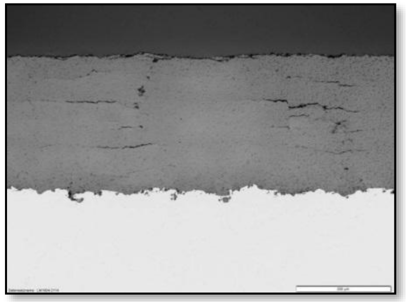
LAwave® – Möglichkeiten und Anwendungsbeispiele der laserakustischen Oberflächenwellen -Spektroskopie

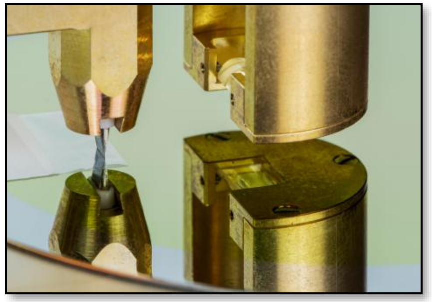
—

Dr. Stefan Makowski
Fraunhofer Institute for Material and Beam Technology IWS, Germany





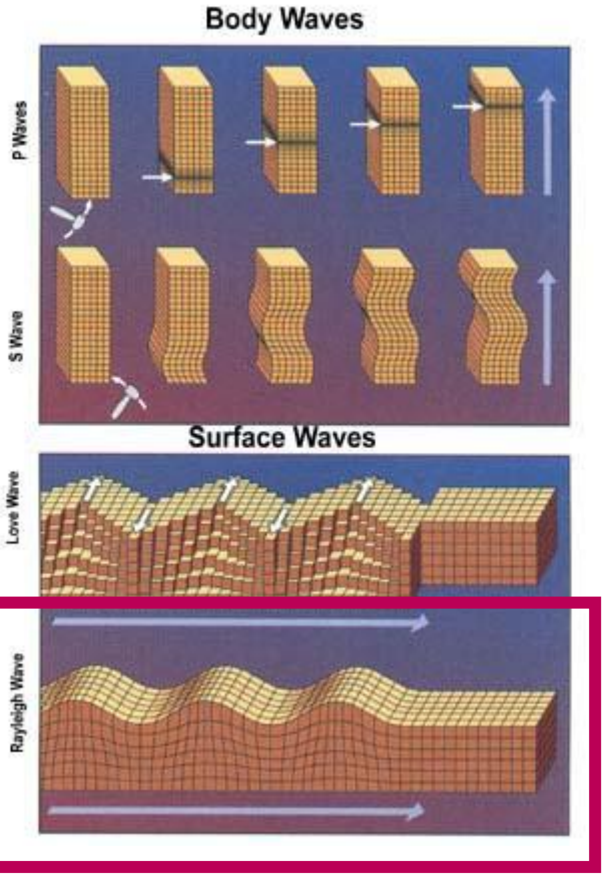




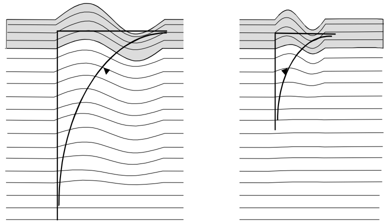
Contents

Introduction

Laser-induced Surface Acoustic Wave Spectroscopy

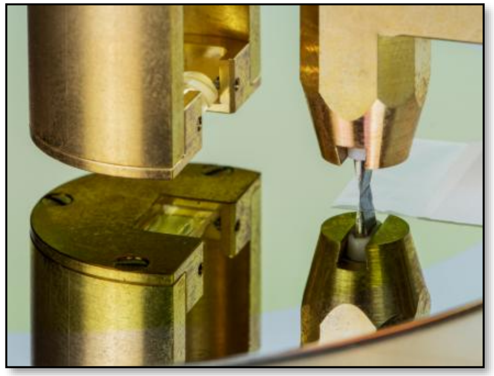


Wikipedia: “Seismic waves”, public domain

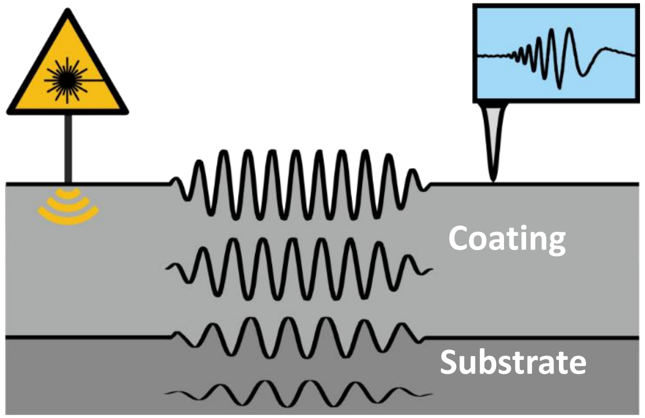


Low frequency

High frequency

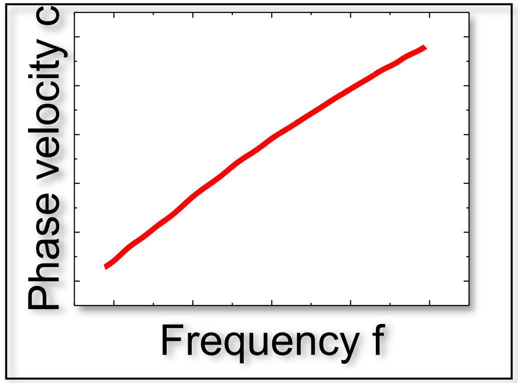


„Miniature earthquake machine“



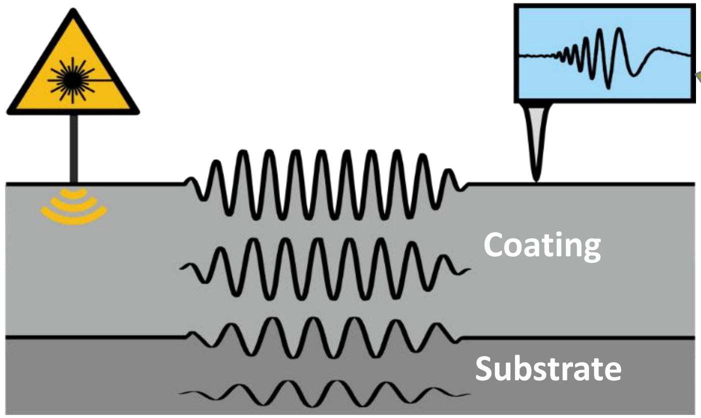
- Young‘s Modulus
- Coating thickness
- Density
- Depth of: Nitriding layer, Case-hardening, Damage layers
- Porosity, crack density
- Delamination
- Build-up structure

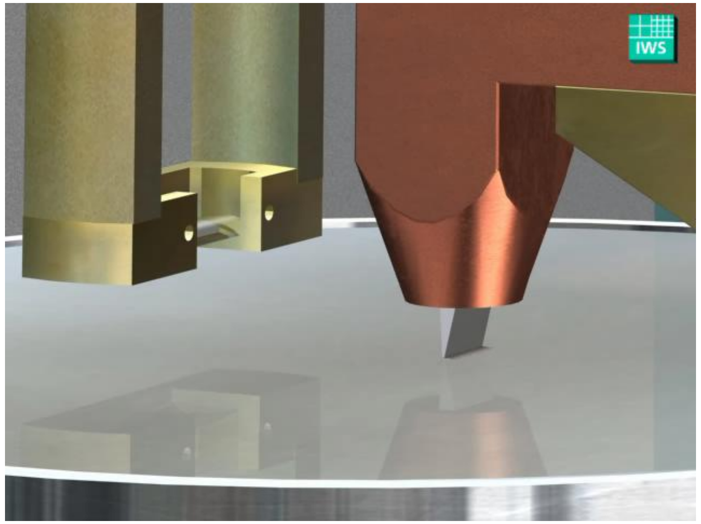
…. Anything that affects your mechanical integrity of the material

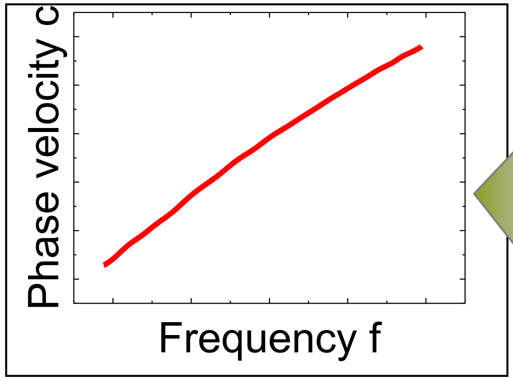


Laser-induced surface acoustic wave spectroscopy - LAwave

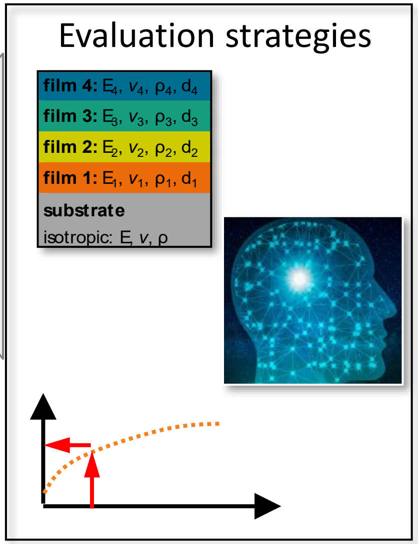
1 2 3 4 5







"Acoustic finger print"



(1) Short laser pulse
→ broadband surface acoustic waves

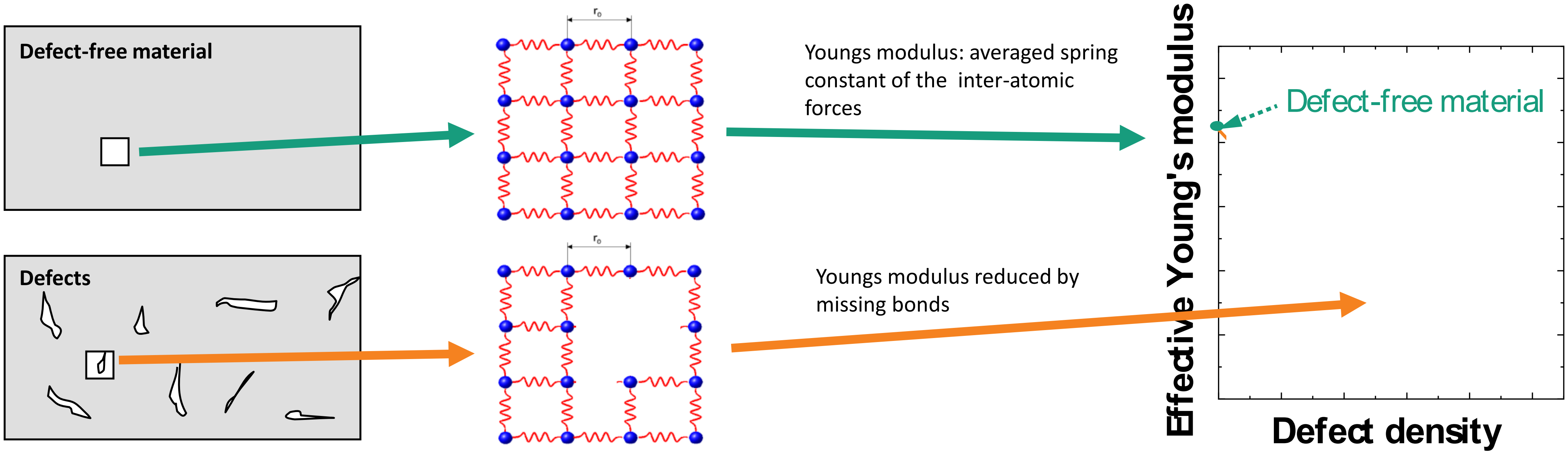
(2) SAW propagation,
depth depends on frequency

(3) SAW measurement: piezoelectric element

(4) Fourier transformation
→ dispersion curve (*c* = *f(f)*) + other data

LAwave setup for small parts
© J. Jeibmann/ Fraunhofer IWS

Effective Young‘s modulus of heterogeneous materials



Defects reducing Young‘s modulus

- Porosity (thermal spraying, AM)
- Integrity, cracks (damage layers, bad adhesion)
- Structure (columnar growth, 3D printing)

Applications

Application - Overview

Coatings

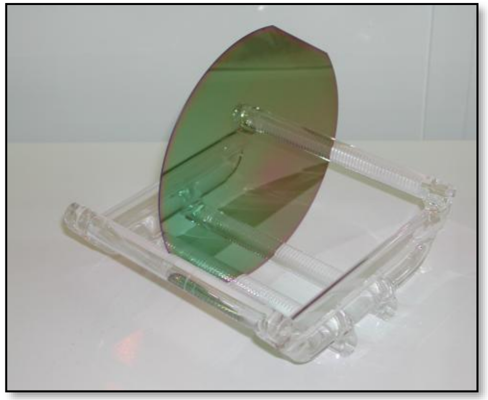
- Thin coatings: PVD, CVD, ALD, spin coating, electroplating, …
- Thick coatings: thermal-spraying, laser-cladding, …
- Metal films
- Polymeric sensor films

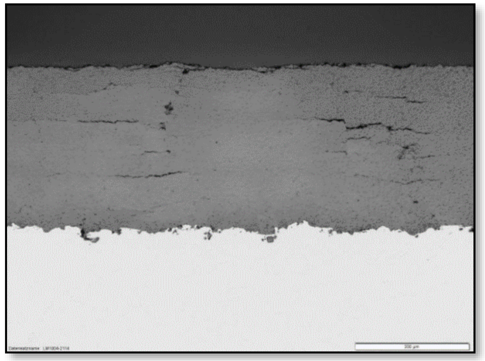
Semiconductors

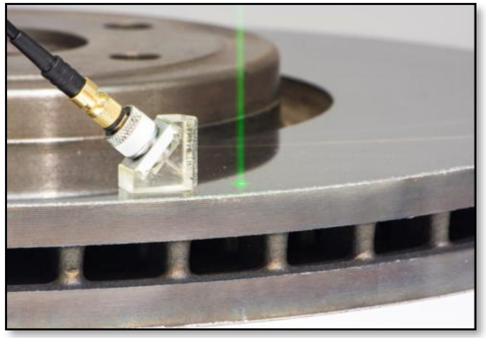
- Wafer bonding layers; damage depth
- Low-k films

Bulk and surfaces

- Case hardening, nitriding
- Rolling, hammering, shot peening
- 3D printing
- Composition (e.g. carbide content in hard metal)







Case study: Very thin films < 10 nm

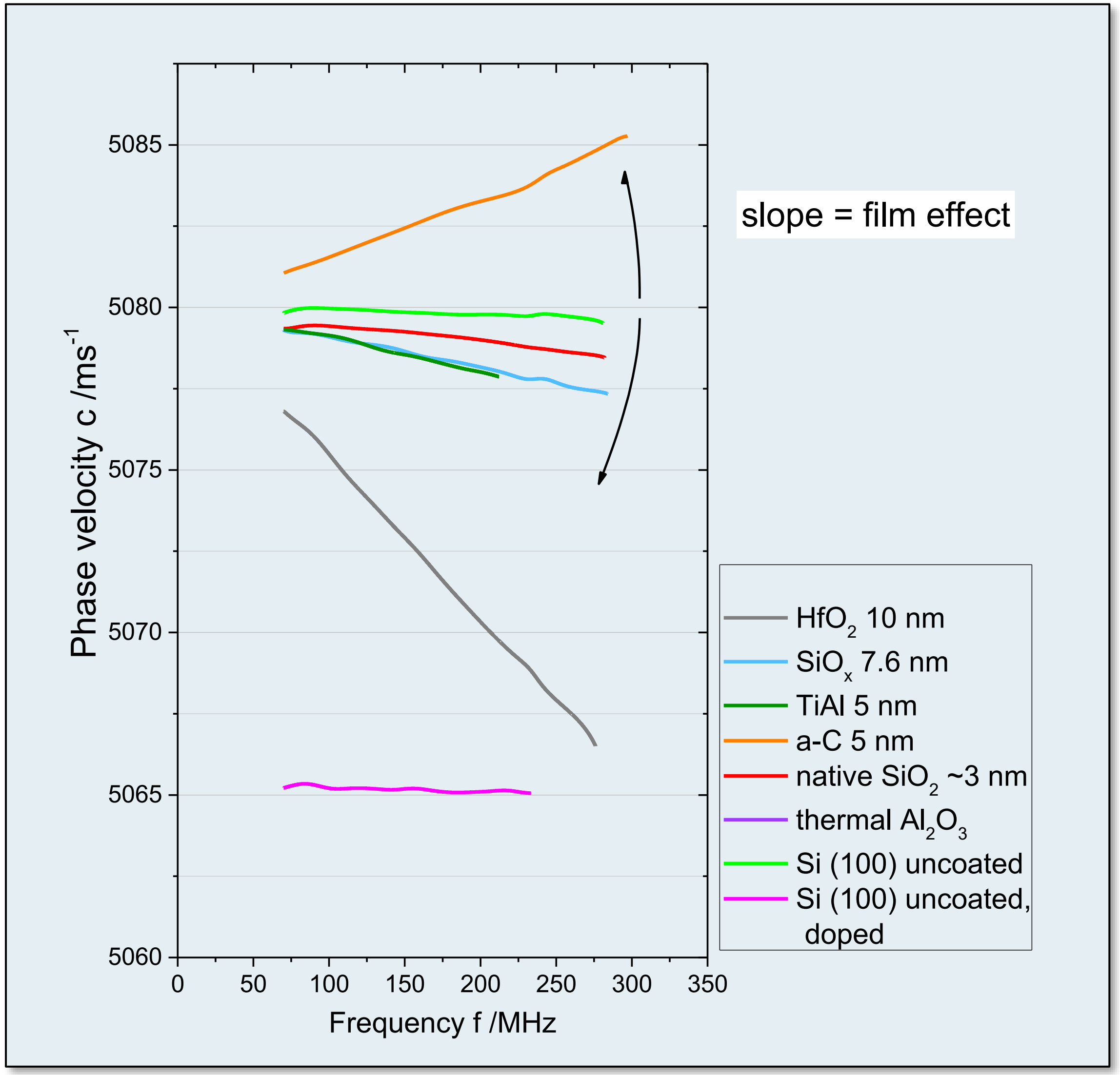
Material

- PVD coatings with thickness < 10 nm

Results

- Measurement of Young's Modulus
  - HfO_2 220.4 GPa
  - Native SiO_2 39.8 GPa
  - SiO_x 41.7 GPa
  - a-C 373.4 GPa
  - TiAlN 142.8 GPa
  - Silicon wafer 165.2 GPa (C11)
  - Silicon wafer (high doping) 162.9 GPa (C11)
- Measurement of thickness of $Si/Al/Al_2O_3$ multilayer stack
  - Thermal Al_2O_3 3.9 nm

➔ Possibilities beyond conventional nanoindentation



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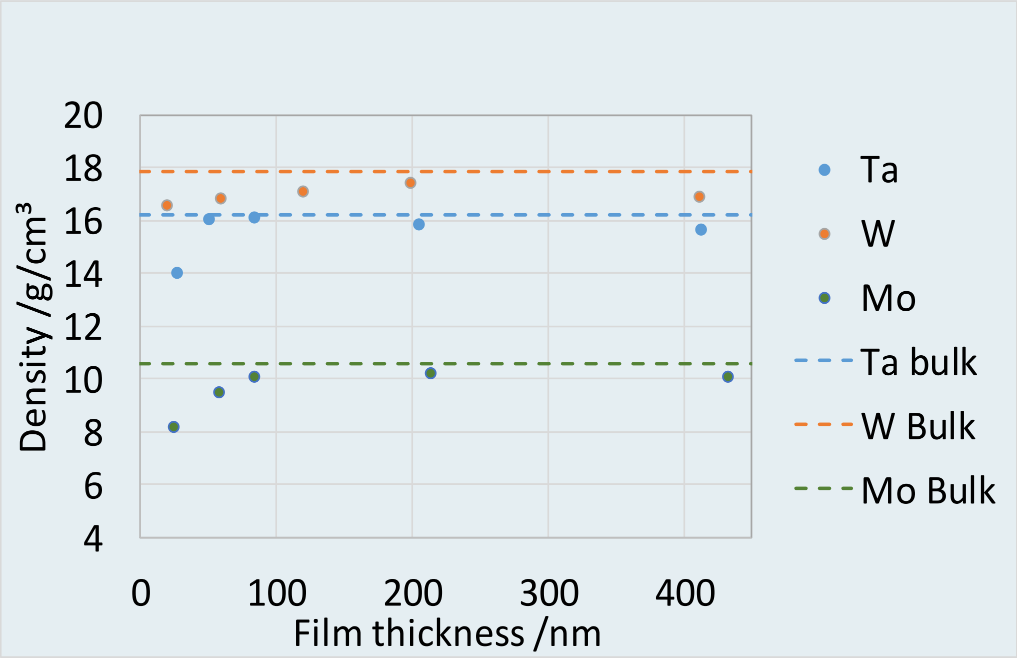
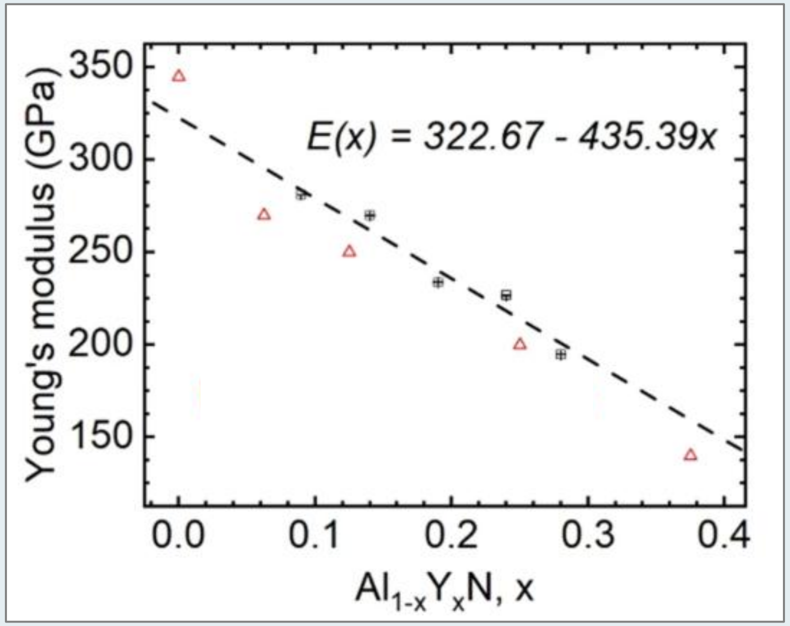
Case study: Structural changes in thin films

Growth dependent density of RF-sputtered refractory metals



T.A. Yakupov, et al., Materials Today: Proceedings 4 (2017) 4469–4476.

Influence of element concentration on Young's modulus in AlYN wurtzite thin films



D. I. Solonenko, et al., Applied Physics A 131,690, (2025)

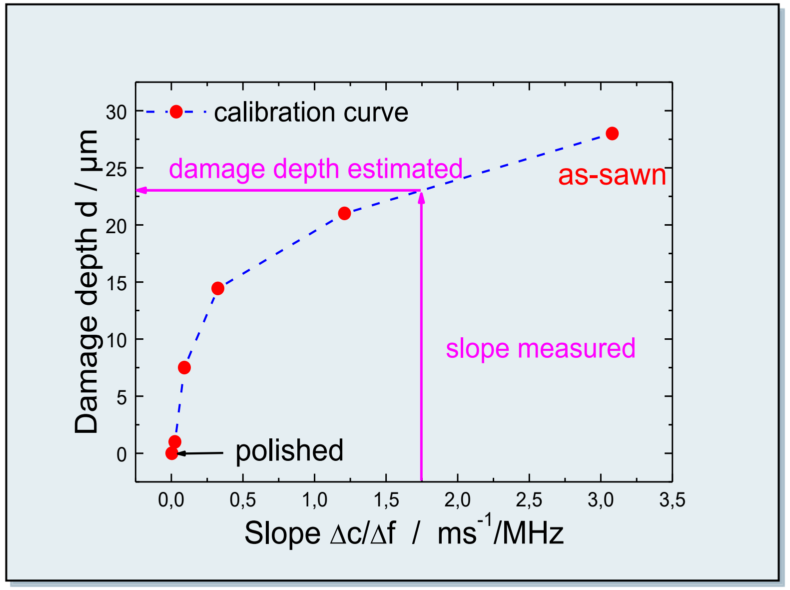
Case study: Subsurface damage in semiconductor wafers

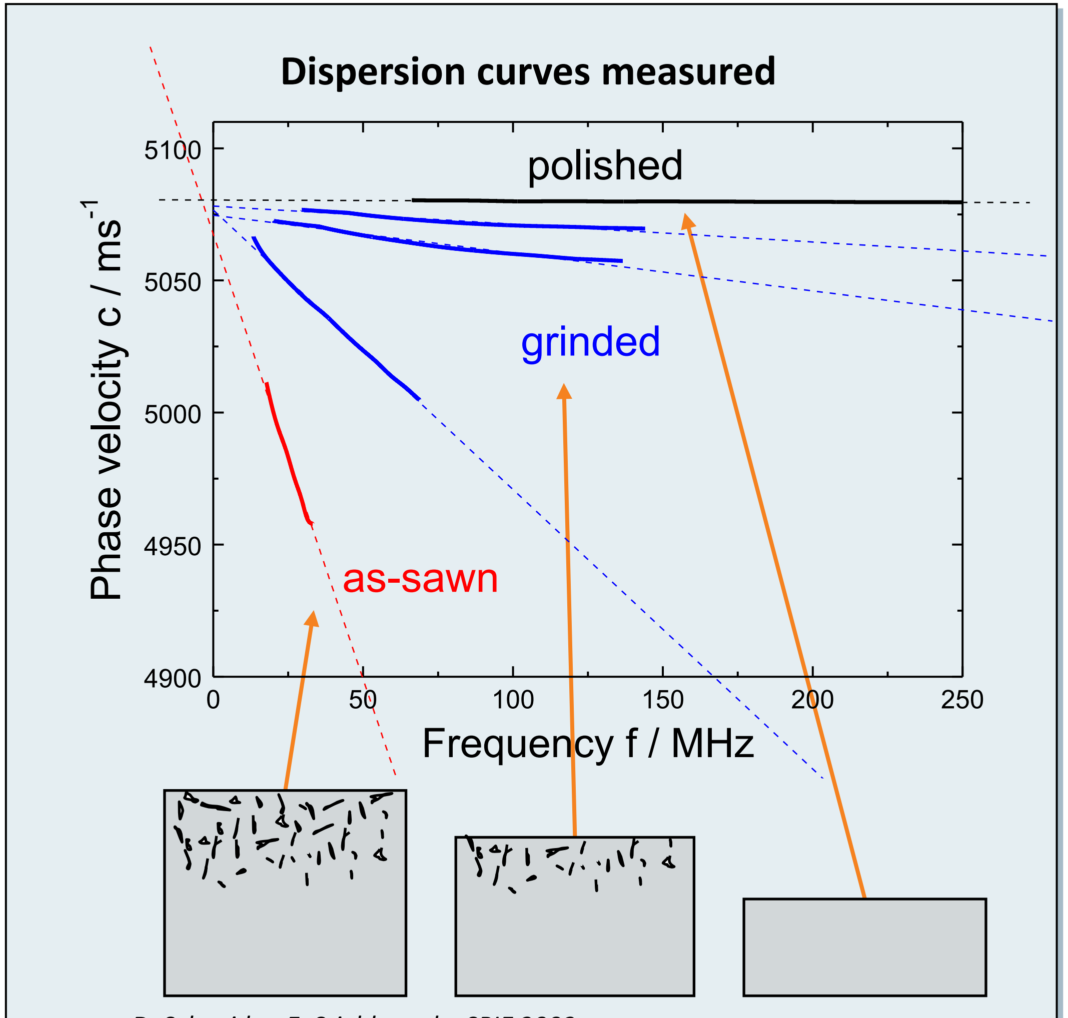
Material

- Semi-conductor surfaces, damaged from processing

Results

- Damage layer ➔ dispersion
- Slope = damage layer depth ➔ allows quantification





D. Schneider, E. Stiehl et. al., SPIE 2002

Case study: Quality control of superhard carbon coatings

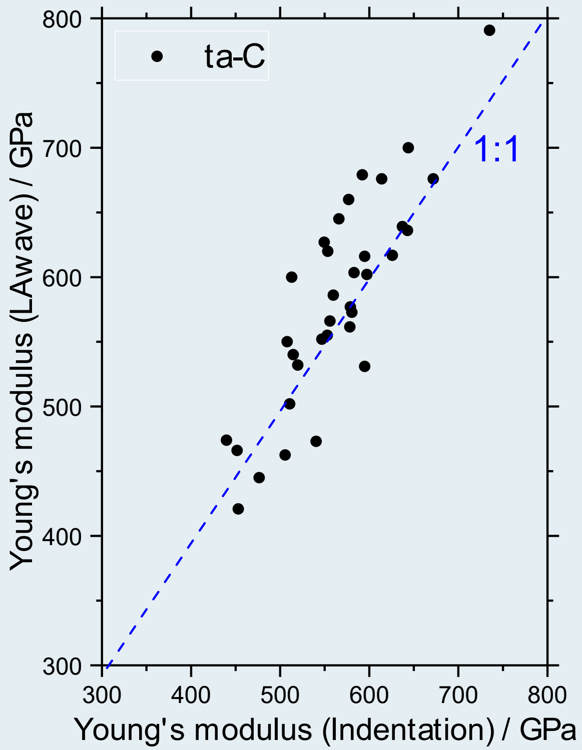
Material

- Superhard amorphous carbon coatings (ta-C, H-free DLC), hardness 40..70 GPa
- Application: Low-wear low-friction coating, e.g. piston pins in ICE, motorcycle chain
- State-of-the art: Nanoindentation → slow and error-prone technique with high indenter wear

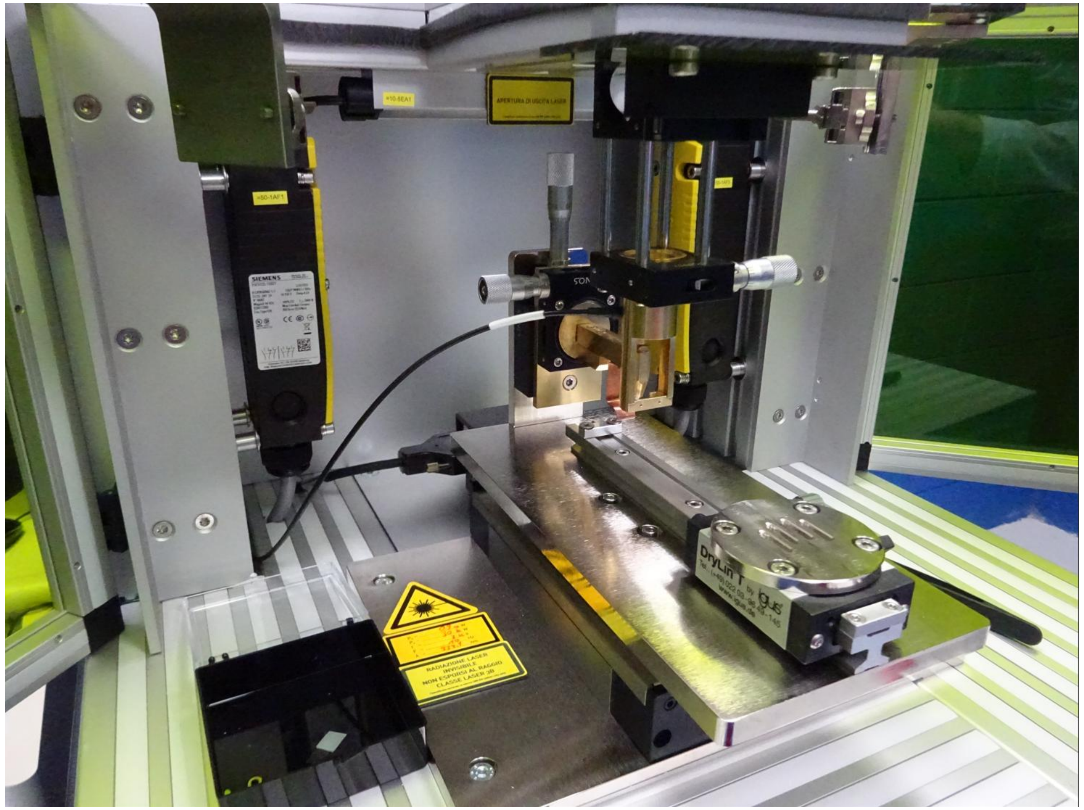
Results

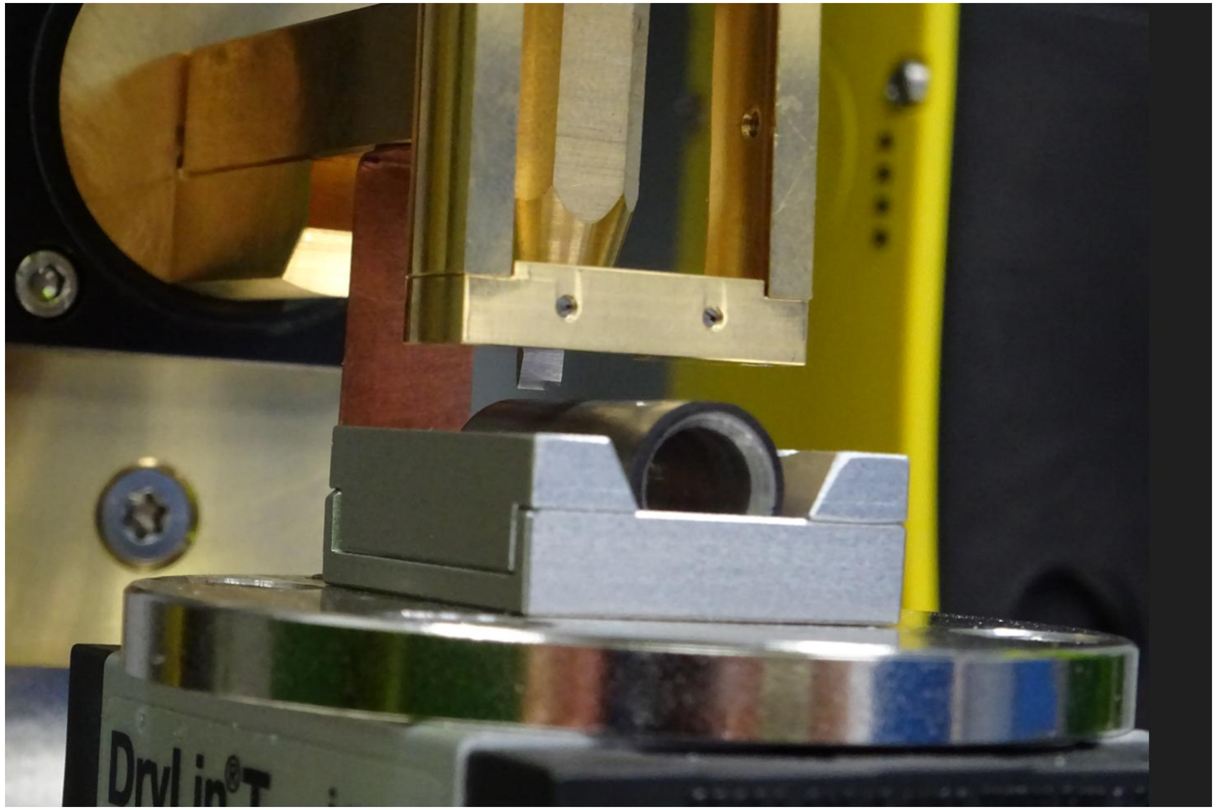
- LAwave allows to access
  - Coating modulus, coating hardness
  - Coating thickness

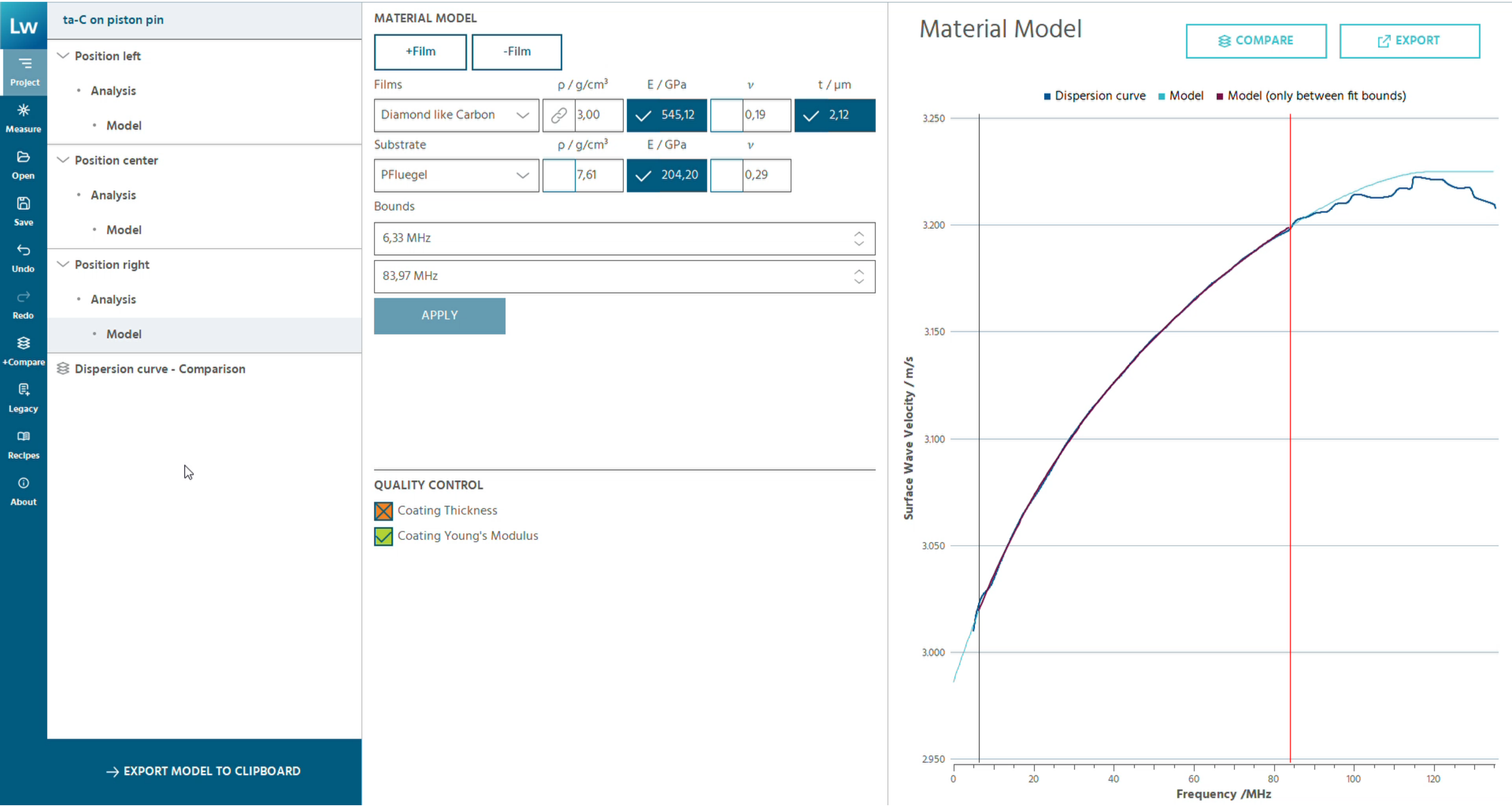
in less than 60 seconds



Case study: Quality control of superhard carbon coatings II

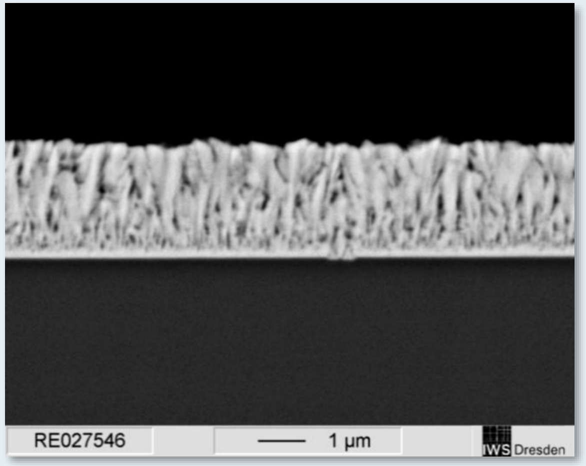




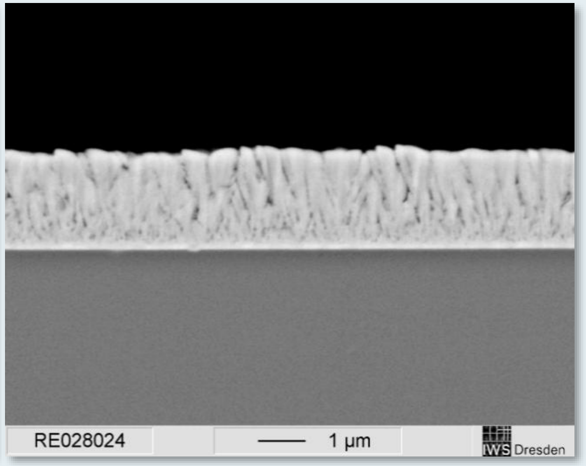

Lw
ta-C on piston pin
Project
Measure
Open
Save
Undo
Redo
+Compare
Legacy
Recipes
About
Position left
Analysis
Model
Position center
Analysis
Model
Position right
Analysis
Model
Dispersion curve - Comparison
→ EXPORT MODEL TO CLIPBOARD
MATERIAL MODEL
+Film
-Film
Films
ρ / g/cm³
E / GPa
ν
t / µm
Diamond like Carbon
3,00
545,12
0,19
2,12
Substrate
ρ / g/cm³
E / GPa
ν
PFluegel
7,61
204,20
0,29
Bounds
6,33 MHz
83,97 MHz
APPLY
QUALITY CONTROL
Coating Thickness
Coating Young's Modulus
Material Model
COMPARE
EXPORT
Dispersion curve
Model
Model (only between fit bounds)
Surface Wave Velocity / m/s
3.250
3.200
3.150
3.100
3.050
3.000
2.950
0
20
40
60
80
100
120
Frequency /MHz

Example: Pores in metal films

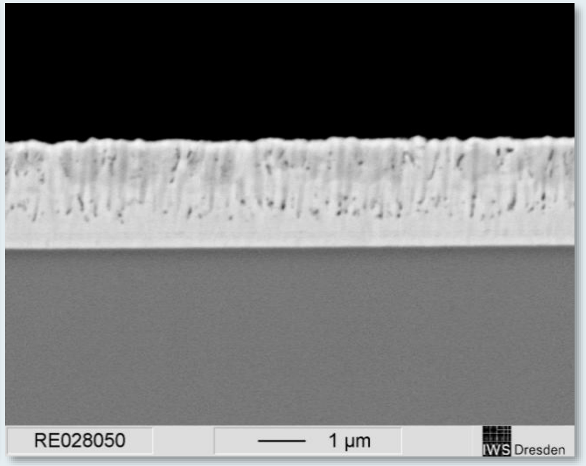
Material: 2 µm Ti coating on Si wafer, deposited with plasma activated electron beam deposition



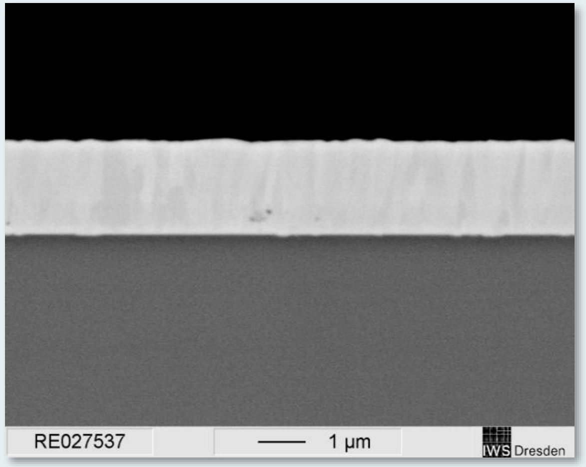
Series 1
E = 68 GPa



Series 2
E = 85 GPa



Series 3
E = 105 GPa



Series 4
E = 113 GPa
(Compact Ti: E = 116 GPa)

M. Leonhardt et. al., Surface and Coatings Technology 185 (2004) 292

➔ **Effective Young's Modulus strongly correlates with porosity observed in SEM cross section**

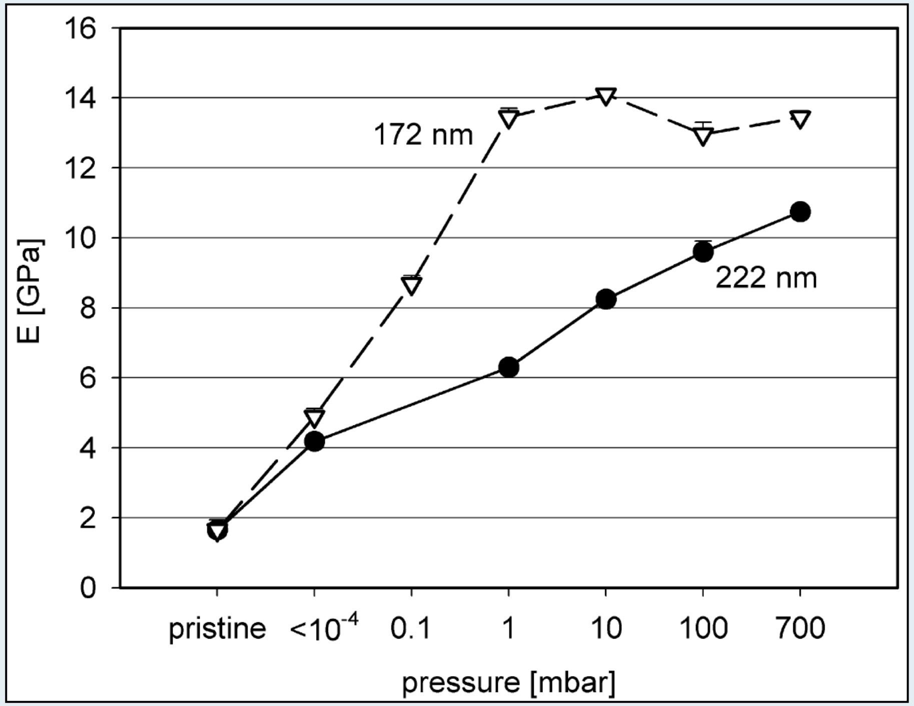
Example: Porous low-k films

Material

- Nano-porous SiCOH low-k films
- High porosity: > 40 %
- Rel. permittivity k < 2.5
- Minimum required stiffness E > 5 GPa

Results

- Young's modulus and density can be measured
- Higher reliability than results from nanoindentation



Irradiation with 172 nm and 222 nm photons, ρ = 1.2 gcm^{-3} and d = 200 nm

Prager et al. Microelectronic Engineering 85 (2008) 2094–2097

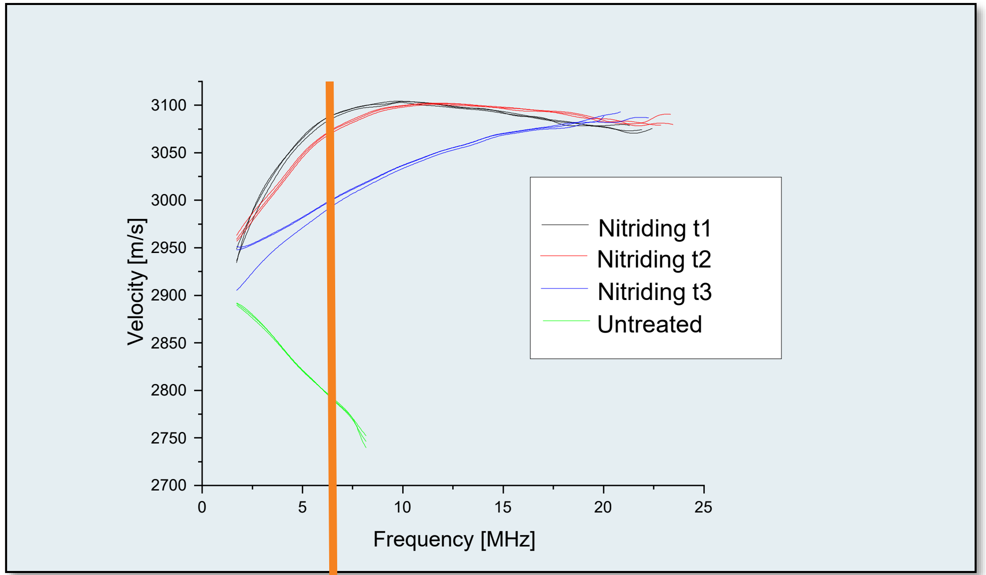
Case study: Nitriding depth in NH_3 reactor

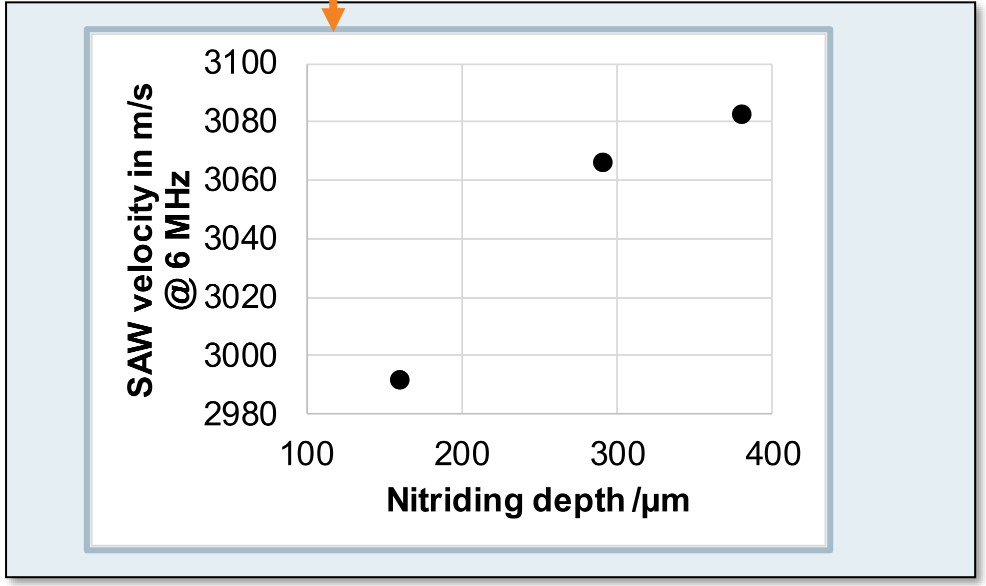
Material

- Steel wall of ammonia reactor
- Nitriding depth depends on processing time and conditions

Application

- Nitriding depth measurement
- Study nitride hardening depth
- Life-time, e.g. in ammonia exposed reactors

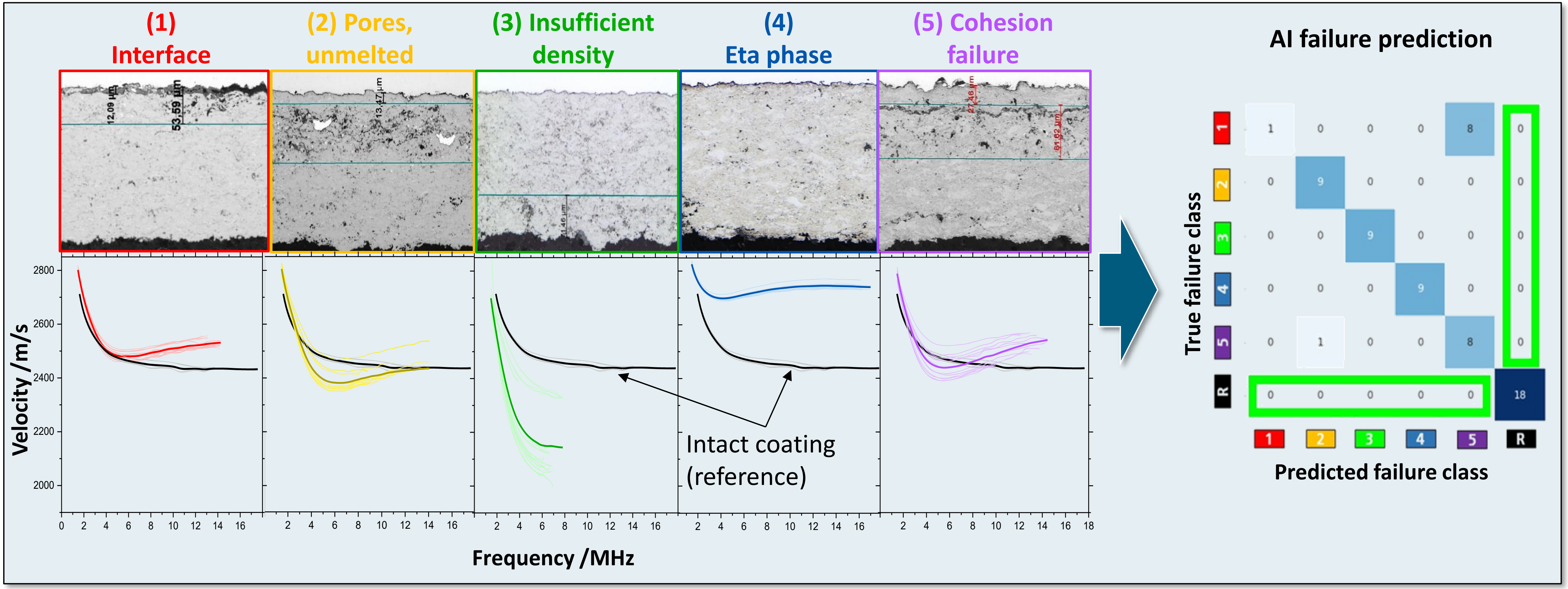




Restricted
LAwave Fraunhofer IWS

Outlook: Identification of defects

WC-Co by thermal spraying



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Methodical Aspects

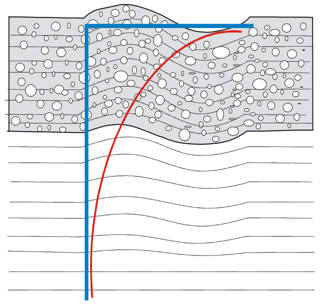
Comparison with instrumented indentation testing

Coating Materials

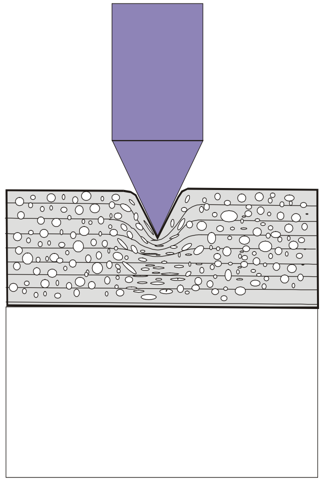
- Porous low-k films

Result

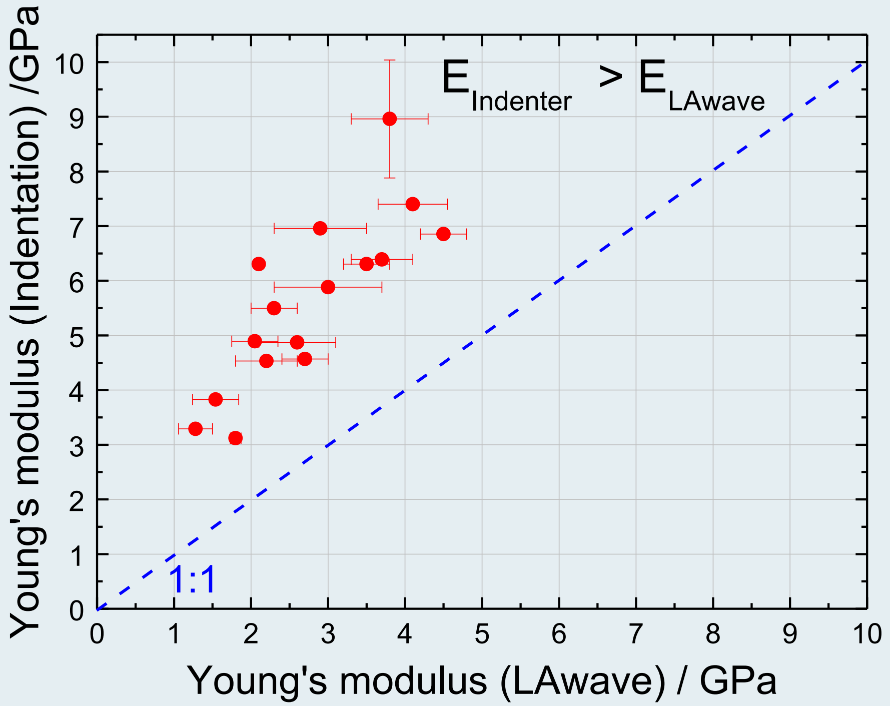
- Effective modulus is strongly overestimated with indentation due to compressed pores



Surface acoustic waves
Reversible deformation
➔ True elasticity



Indentation
Densification of microdefects
➔ Distorted results



S. Frühauf, University of Chemnitz

This example: $E_{Indenter} \approx 2 \cdot E_{LAwave}$

Comparison with instrumented indentation testing (nanoindentation)

	LAwave	Nanoindentation
Method	Dynamic: Sound velocity $c \sim \sqrt{(E/\rho)}$	Quasi-static: $E_r \sim dP/dh$
Measuring area	> 5 x 5 mm² (integral method)	< 10 µm² (local method)
Measuring time	t ~ 1 min	t >> 1 min (incl. preparation and calibration)
Minimal film thickness	d > 1 nm	d > 100 nanometers
Surface roughness	No requirements	Smooth surface necessary
Difficult material systems	Transparent and high damping materials; coated foils	Soft and superhard materials, very thin coatings
Benefits	Thin and porous coatings	True plastic behavior; high lateral resolution

Development and Technology Readiness

State of the art

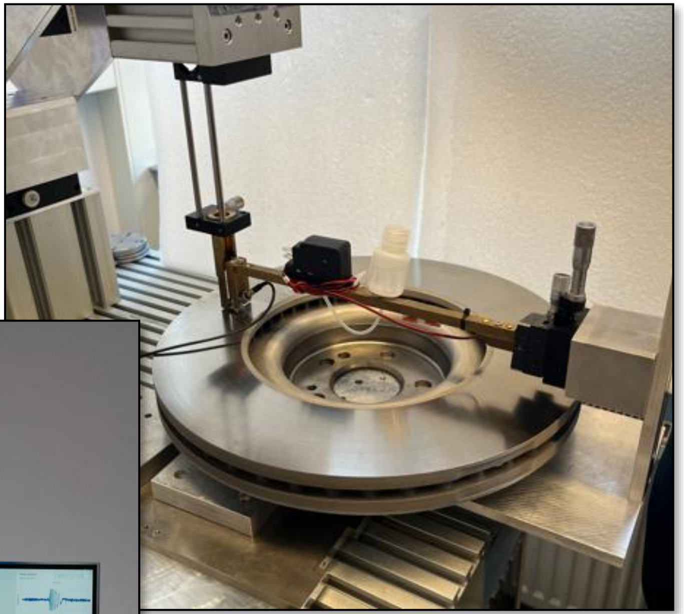
- Standalone system, manual handling, quality control concepts - TRL 9
- 40^+ systems world wide

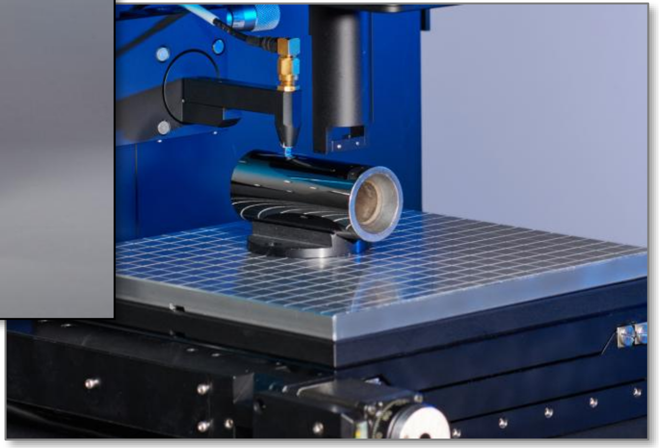
New features under development

- Automated measurement – TRL4
- In-situ measurement up to 600 °C – TRL 4
- AI assisted evaluation – TRL 5
- Mobile head for robot or hand measurement

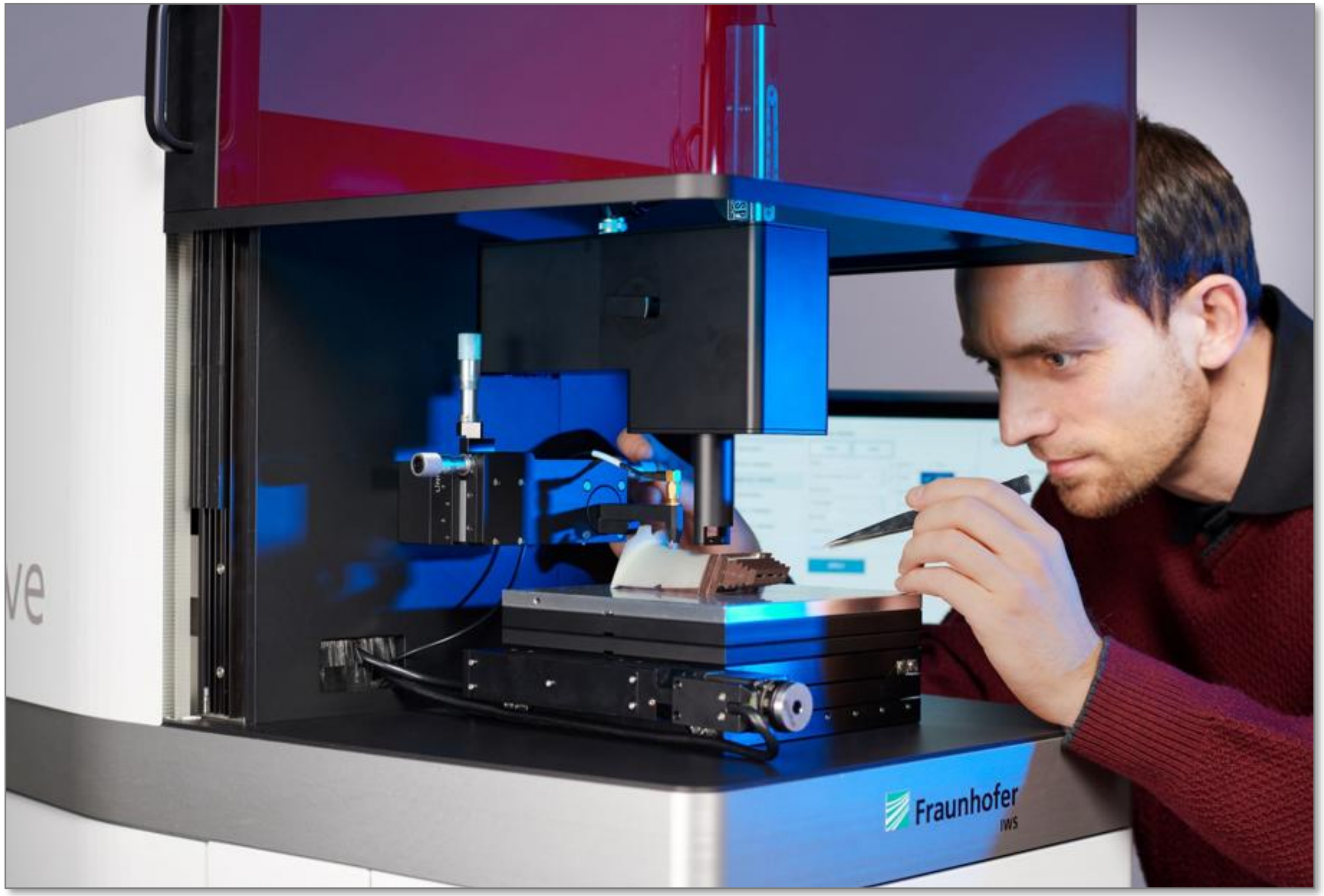
Fraunhofer IWS provides

- 35^+ years of experience in LAwave application across all industries
- Development partnership for industry and academia
- Job measurements, feasibility studies, quality control concepts
- Custom solutions for research, quality control, analysis and automation





Summary



LAwave®

- Mechanical properties of coatings and surfaces
- Fast and non-destructive
- Coatings from 1 nm to 1 mm
- Used for R&D and Quality Control
- 35⁺ years of experience; 40⁺ systems world-wide, 70⁺ peer reviewed contributions

LAwave around the world

Distribution USA

Fraunhofer USA
44792 Helm Street
Plymouth, MI 48170 USA
Phone: +1 734-354-4335
lawave@fraunhofer.org

Headquarter Germany

Fraunhofer Institute for Material and Beam Technology IWS
Winterbergstrasse 28, 01277 Dresden, Germany
Dr. Martin Zawischa
+49 351 83391 3096
lawave@iws.fraunhofer.de

Distribution Japan

Sanyo Trading Co., Ltd.
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101-0054 Japan
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Distribution China

GermanTech Nano Co. Ltd.
Room 1408, Tower B, Techart Plaza
No.30 Xue Yuan Road, Haidian District,
100083 Beijing, P.R.China
+86 10 82867920/21/22
contact@germantech.com.cn

Contact

lawave@iws.fraunhofer.de

Dr. Martin Zawischa +49 351 83391 3096
Dr. Stefan Makowski +49 351 83391 3192

Fraunhofer Institute for Material and Beam Technology IWS
Winterbergstraße 28
01277 Dresden
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Scientific Literature using Lawave: Link

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