# Atomic force microscopy – what is it all about, and what does it tell us about the microstructure of metals?

**Sigrun Hirsekorn**
Fraunhofer Institute for Nondestructive Testing (IZFP)
Campus E3.1, 66123 Saarbrücken, Germany

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# Outline

- Motivation
- Atomic Force Microscopy (AFM)
  - Principle, conventional (quasi-static) operation modes
  - AFM sensors
  - Dynamic operation modes, especially in the ultrasonic frequency range
- Application to metals
  - Nanostructured (nanocrystalline) nickel
  - TWIP (twinning induced plasticity) and unalloyed steels
- Summary

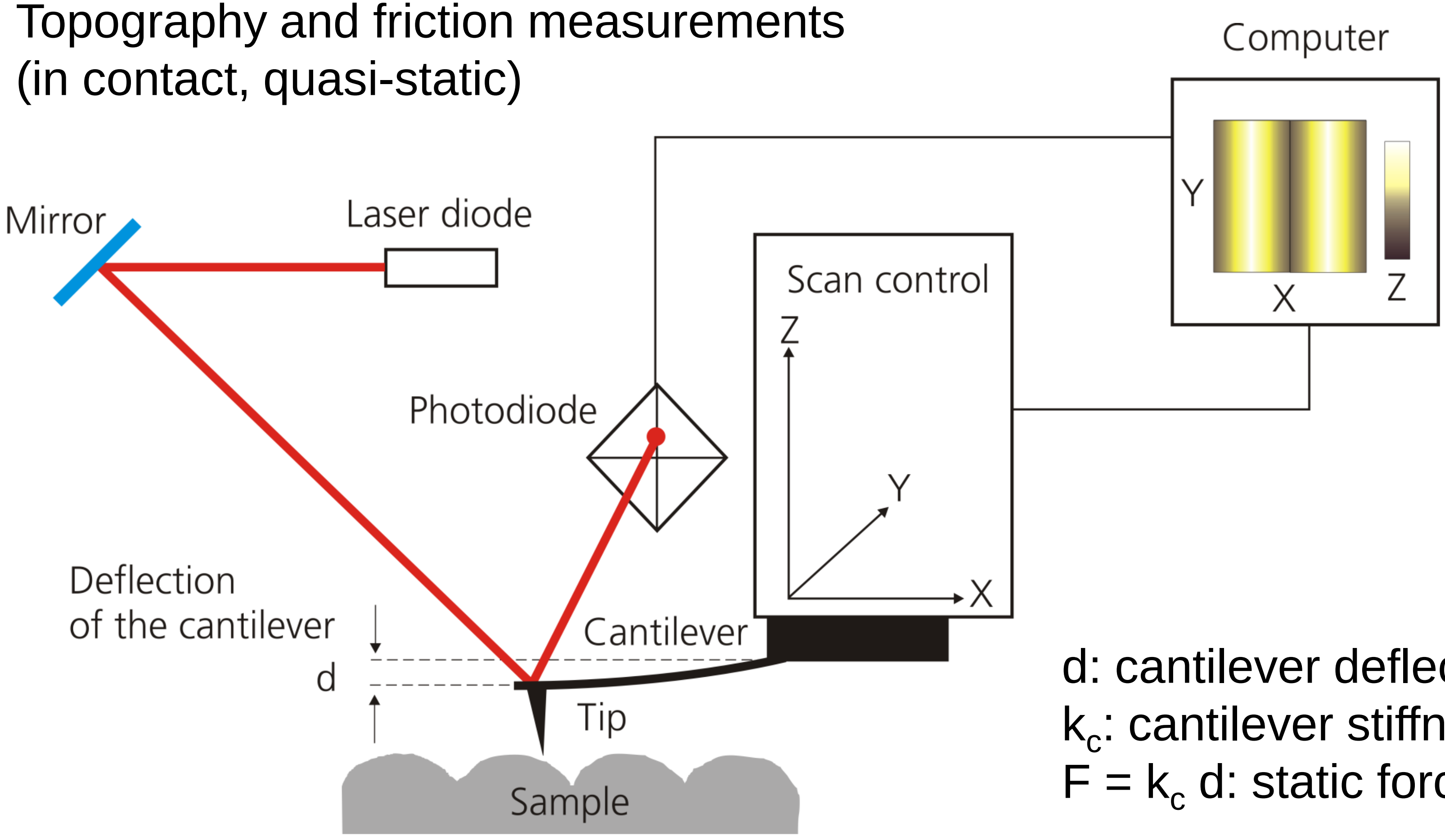
# Motivation

- Different applications require different materials (properties)
- Macroscopic elastic and plastic properties of materials (metals) depend on their microstructure, i.e. good prospects for materials design by selective adjustment of microstructure
- Nanocrystalline and/or interface dominated materials
  - Grain size and shape and their distributions
  - Secondary phase inclusions and/or precipitates and their size and shape distributions
- The development of new steel grades:

  Common optimization of contradicting properties as e.g. weight reduction and formability versus high stiffness and strength
- Nondestructive characterization techniques to image structures and probe material properties on the micro- and nanoscale

# Principle of Atomic Force Microscopy (AFM)

Topography and friction measurements
(in contact, quasi-static)

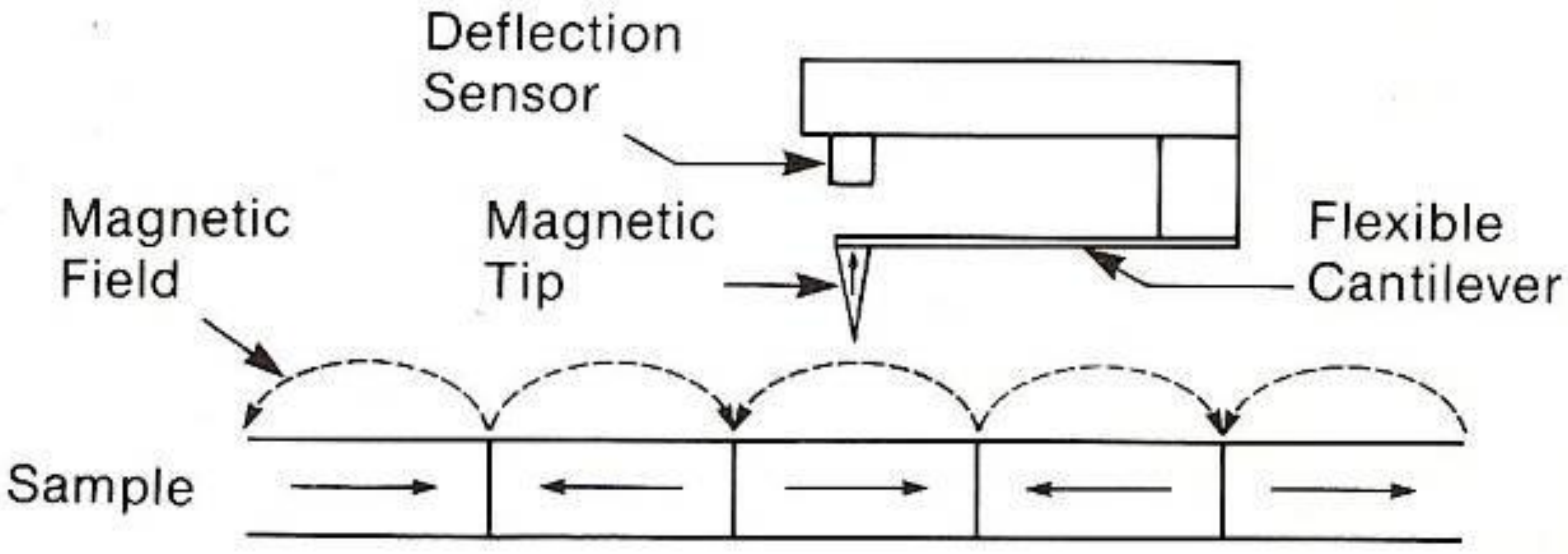



d: cantilever deflection
$k_c$: cantilever stiffness
$F = k_c$ d: static force

G. Binnig, C.F. Quate, and C. Gerber, Phys. Rev. Lett. 56, p. 930 (1986)

# Principle of Magnetic Force Microscopy (MFM)

Magnetic sample, cantilever tip coated with a ferromagnetic material
⇒ Mapping of magnetic domain structures at the sample surface by recording the force gradient between sample and tip
(in force contact, quasi-static)

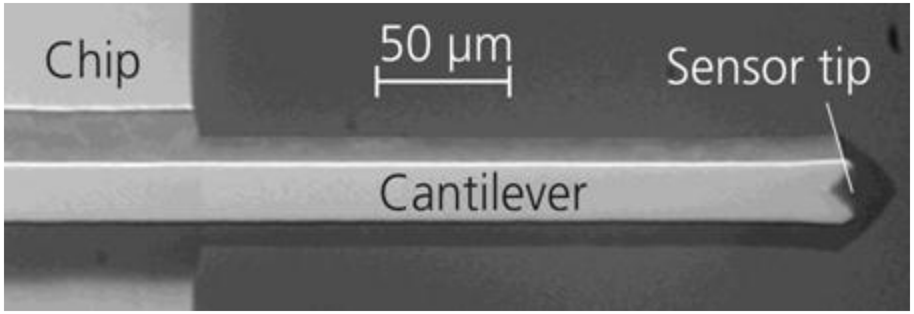



Y. Martin and H.K. Wickramasinghe, Appl. Phys. Lett. 50 (1987) 1455
P. Grütter et al. in: Scanning Tunneling Microscopy II, 1992

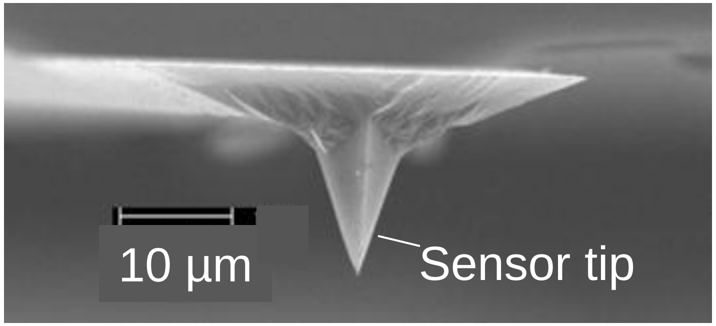
# Sensors of commercial atomic force microscopes (AFM)

Small elastic beams:
Length: a few 100 µm, width: a few 10 µm, thickness: a few µm,
Sensor tip: Radius of a few nm up to of a few 100 nm,
Material: Silicon single crystal (cantilever and chip one piece),
Vibration resonances in the ultrasonic frequency range,
i.e. usable as a near field ultrasonic probe.

Optical microscopy image

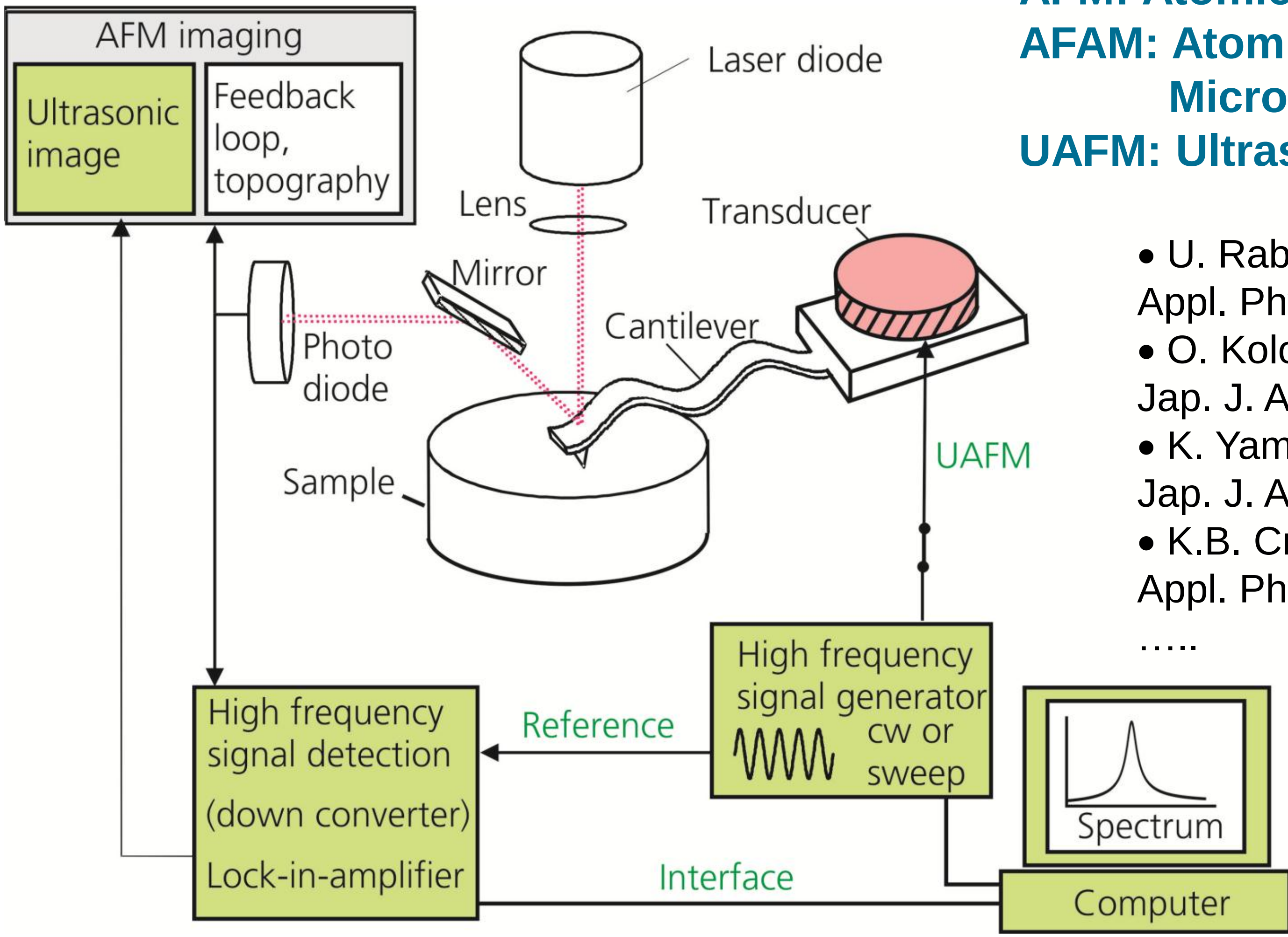



SEM image




Typical values:
1. Free bending resonance frequency: 10 - 300 kHz
Spring constant: ca. 0.1 - 60 N/m

# AFAM and UAFM set-up




**AFM: Atomic Force Microscopy**
**AFAM: Atomic Force Acoustic Microscopy**
**UAFM: Ultrasonic AFM**

- U. Rabe and W. Arnold, Appl. Phys. Lett. 64 (1994) 1493
- O. Kolosov and K. Yamanaka, Jap. J. Appl. Phys. 32 (1993) 22
- K. Yamanaka and S. Nakano, Jap. J. Appl. Phys. 35 (1996) 3787
- K.B. Crozier et al. Appl. Phys. Lett. 76 (2000) 1950

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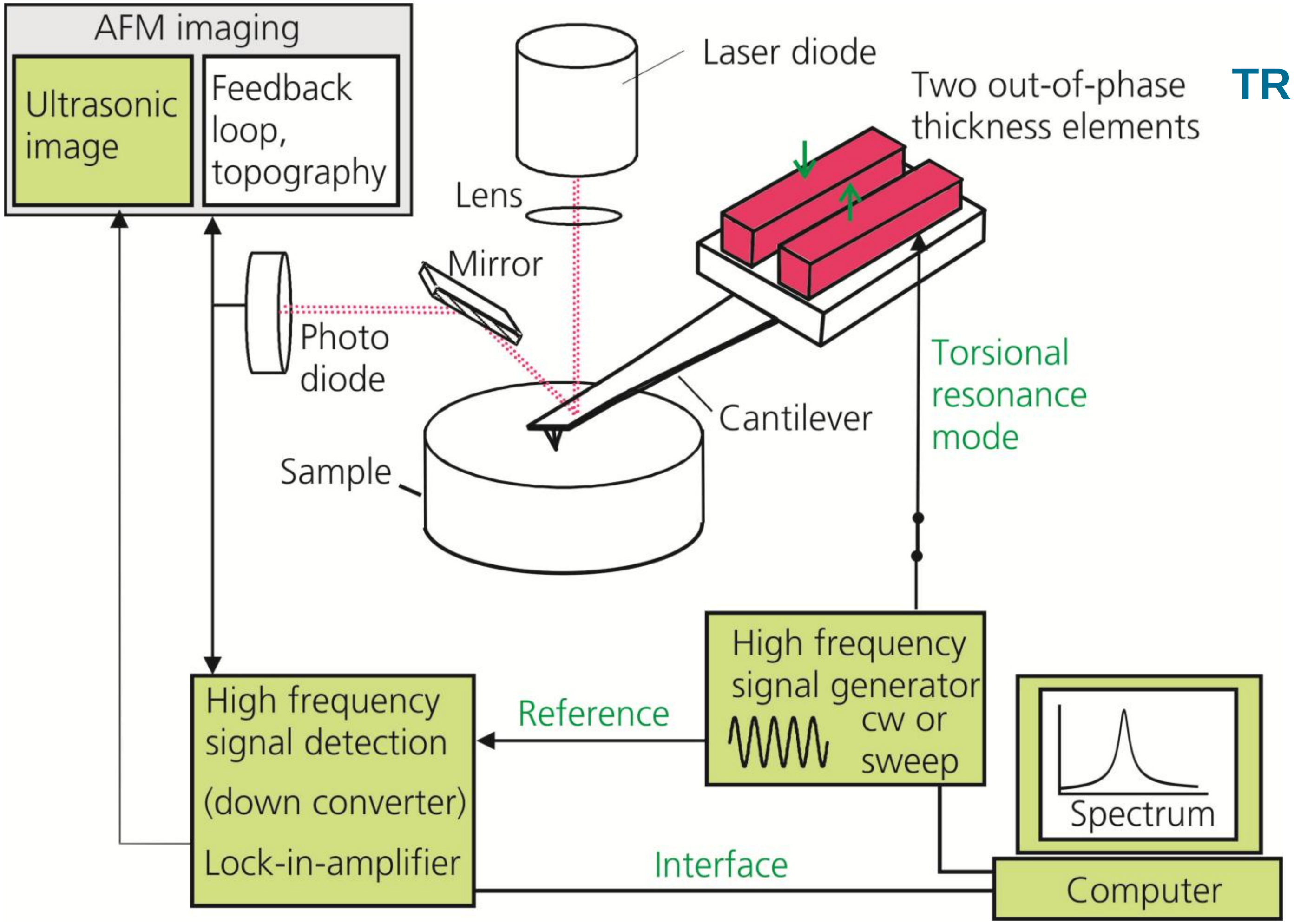
# Lateral AFAM and TR-mode set-up

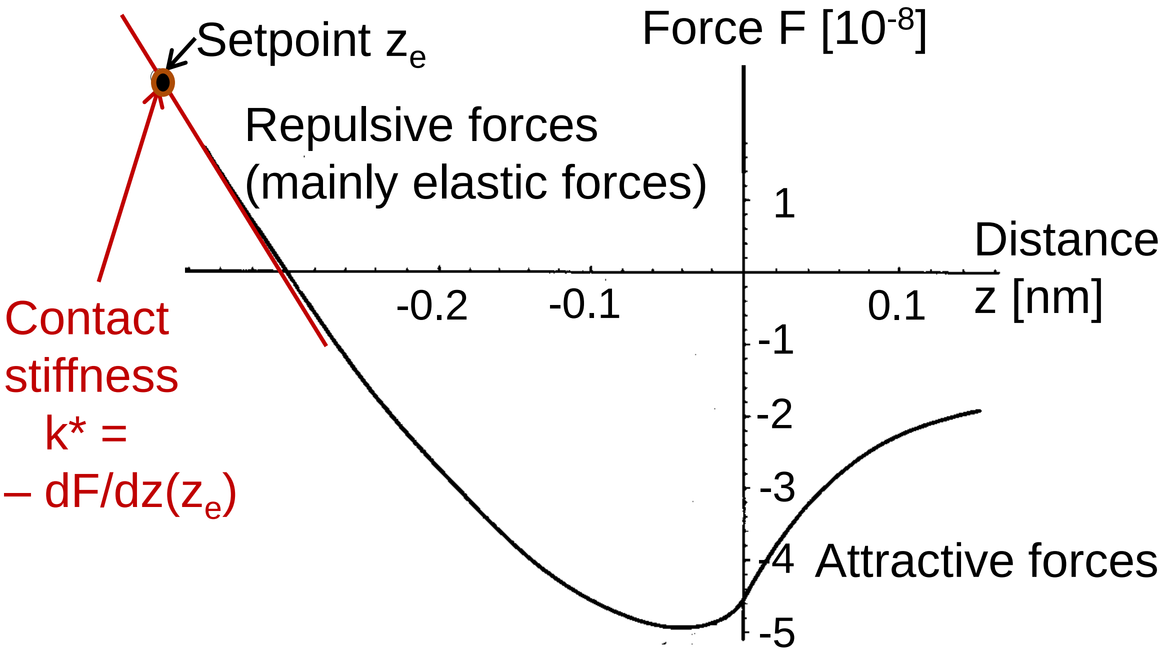



**AFAM: Atomic Force Acoustic Microscopy**
**TR: Torsional resonance**

- A. Caron, U. Rabe, M. Reinstädtler, J.A. Turner, W. Arnold, Appl. Phys. Lett. 85 (2004) 6398
- M. Reinstädtler, T. Kasai, U. Rabe, B. Bhushan, W. Arnold, J. Phys. D: Appl. Phys. 38 (2005) R269
- V. Scherer, M. Reinstaedtler, W. Arnold, Eds. B. Bhushan et al., Applied Scanning Probe Methods (2003) 75-115

…..

# Quasi-static vertical tip – sample interaction force curve

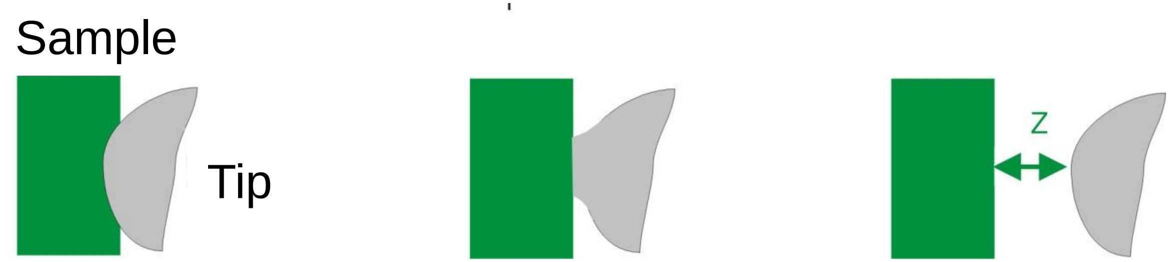



Chromium sample, Silicon cantilever;

Force curve calculated from a measured AFM - force - distance curve

AFM operation modes, quasi-static or dynamic:
- Contact
- Intermediate contact
- Non-contact (at least force contact)

Dynamic:
- Intermittent contact




S. Hirsekorn, Nanotechnology 8 (1997) 57-66

# Dynamic AFM operation modes, non-resonant

- Force modulation mode, contact, operation frequencies below the cantilever resonances, small amplitude excitation at the cantilever chip, evaluation of changes in the cantilever deflection amplitude by the local sample stiffness
- Pulsed force mode (PFM), intermediate-contact, operation frequencies below the cantilever resonances, large amplitude excitation at the cantilever chip covering the complete force-distance-curve
- Piezo mode (PM), contact, operation frequencies below the cantilever resonances, conducting cantilever and tip, piezoelectric sample, exploits locally the piezoelectric effect
- Scanning local acceleration microscopy, contact, ultrasonic operation frequency not in cantilever resonance, evaluation of changes in quasi-static cantilever deflection, excitation of the sample
- Ultrasonic force microscopy (UFM), contact, ultrasonic operation frequency not in cantilever resonance, evaluation of changes in quasi-static cantilever deflection, excitation at the cantilever chip



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# Dynamic AFM operation modes, resonant

- Tapping mode (TP), intermittent contact, ultrasonic operation frequencies at a free cantilever resonance, excitation at the cantilever chip, evaluation of changes in the cantilever vibration amplitude or phase caused by the short contact of the tip with the sample during each vibration cycle, measurement of topography

- Contact resonance AFM, ultrasonic operation frequencies at or close to a contact resonance of the cantilever

# Contact resonance AFM

- Atomic Force Acoustic Microsopy (AFAM), vertical and lateral
- Ultrasonic Atomic Force Microscopy (UAFM)
- Torsional resonance (TR) mode
- Ultrasonic Friction Force Microscopy (UFFM)
- Ultrasonic Piezo-Mode (UPM), piezoelectric samples

exploit flexural or torsional vibration resonances of AFM cantilevers while its sensor tip is in sample surface contact;
lateral resolution: nm range depending on the contact radius

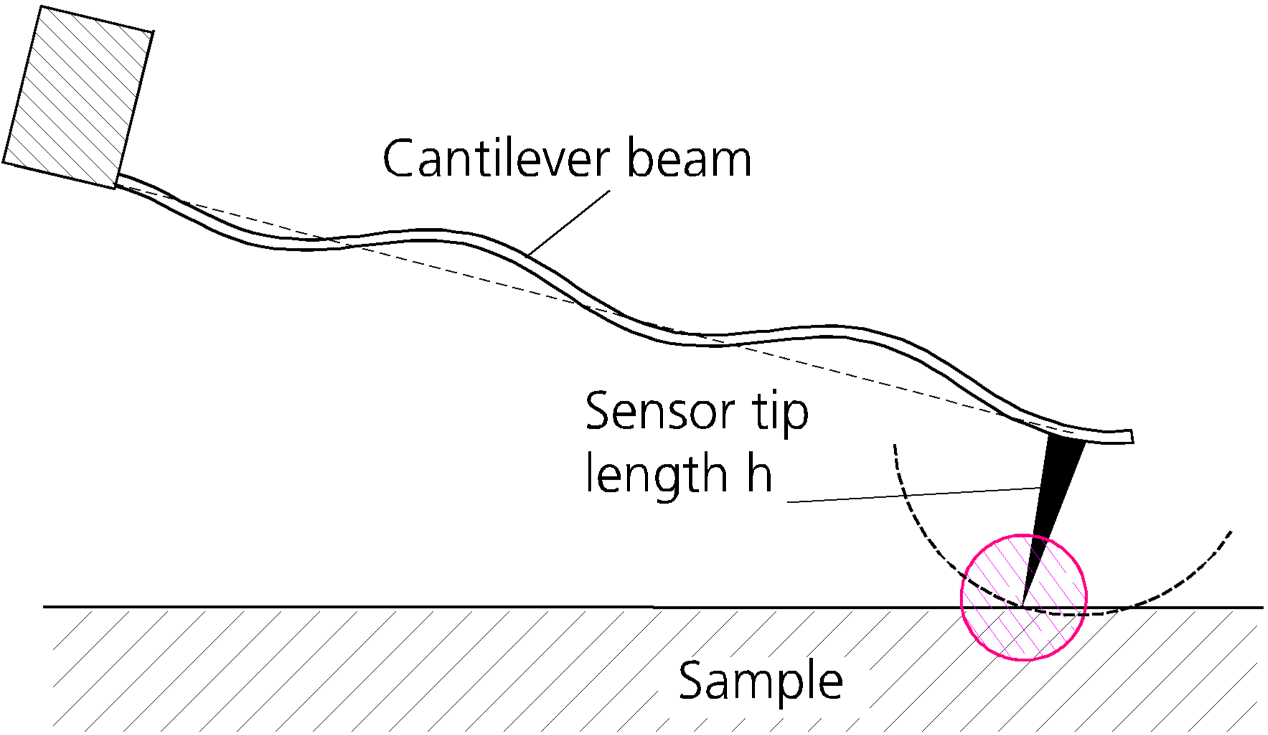
Imaging of sample surfaces:

- Amplitude
- Phase
- Contact resonance frequency

Spectroscopy:

Contact resonance spectroscopy (Quantitative determination of local material parameters)

# Flexural vibrations of a beam of constant cross section




Equation of motion of flexural vibrations in a bar:

$$EI\frac{\partial^4 y}{\partial x^4} + \gamma \frac{\partial y}{\partial t} + \rho A \frac{\partial^2 y}{\partial t^2} = 0$$

E: Young's modulus
ρ: Mass density
γ: Damping constant
A: Cross section area
I: Area moment of inertia
$k_C$: Static spring constant of the AFM cantilever beam

Boundary conditions:
Clamped – free beam ⇒ solutions from textbooks
Clampled – surface coupled beam ⇒ nonlinear force curve



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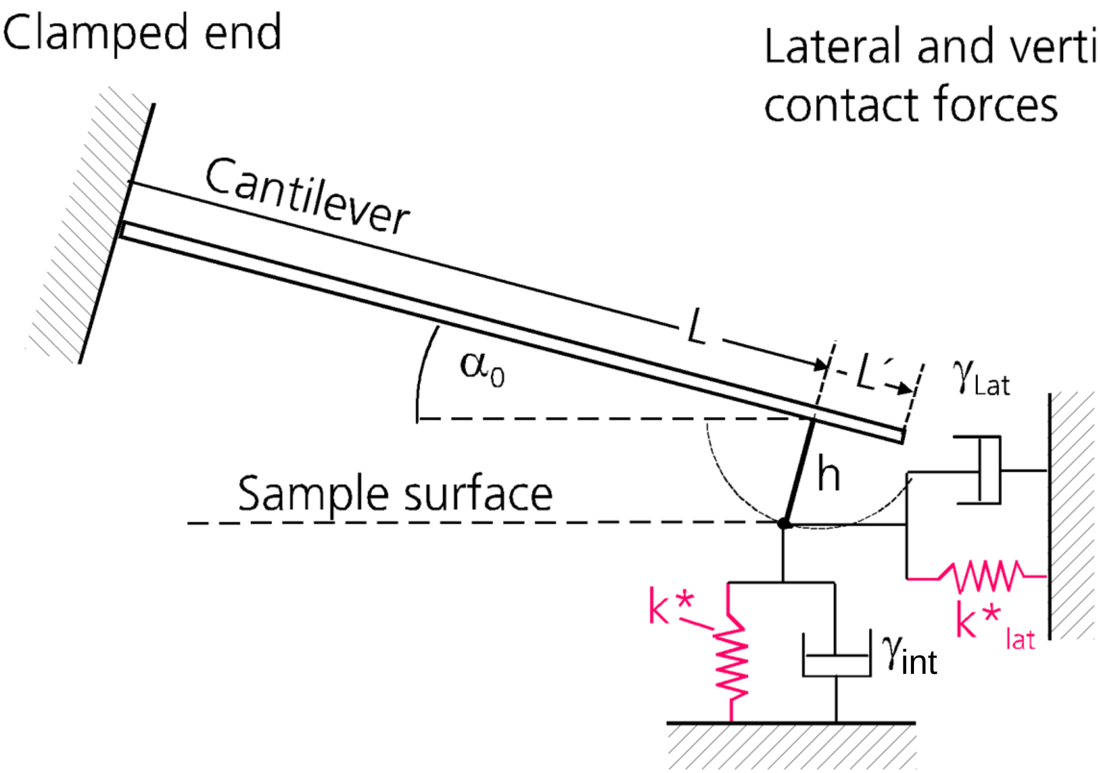
# Cantilever beam and contact model




$k_C$: Static spring constant of the AFM cantilever beam

- k*: Contact stiffness
- $k^*_{lat}$: Lateral contact stiffness
- $\gamma_{int}$: Interaction damping
- $\gamma_{lat}$: Lateral interaction damping
- L: Cantilever length, clamped end - tip
- L´+L: Total cantilever length
- h: Sensor tip height
- $\alpha_0$: Angle between cantilever and sample surface

Repulsive region: Hertzian model of the contact forces
⇒ Relation between the contact stiffness k* and the local indentation modulus of the sample surface

Equivalent for shear forces and torsional resonances ⇒ shear modulus



U. Rabe, Applied Scanning Probe Methods II, Springer, 2006, 37-90

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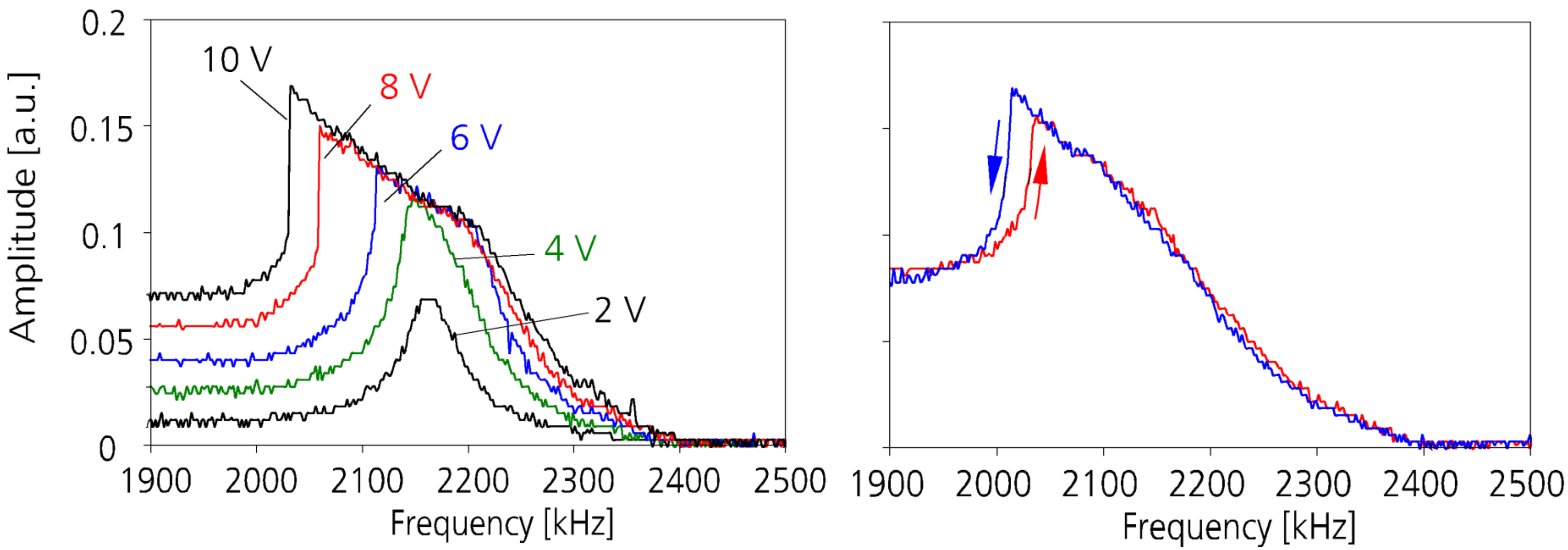
# AFAM contact resonance spectra, vertical

Linear and nonlinear contact forces




Increase of the excitation amplitude: softening effect, asymmetric shape of the resonance peak, amplitude jump

Inversion of the direction of the frequency scan: hysteresis

Si-sample; Si-cantilever, 2nd cantilever contact bending mode



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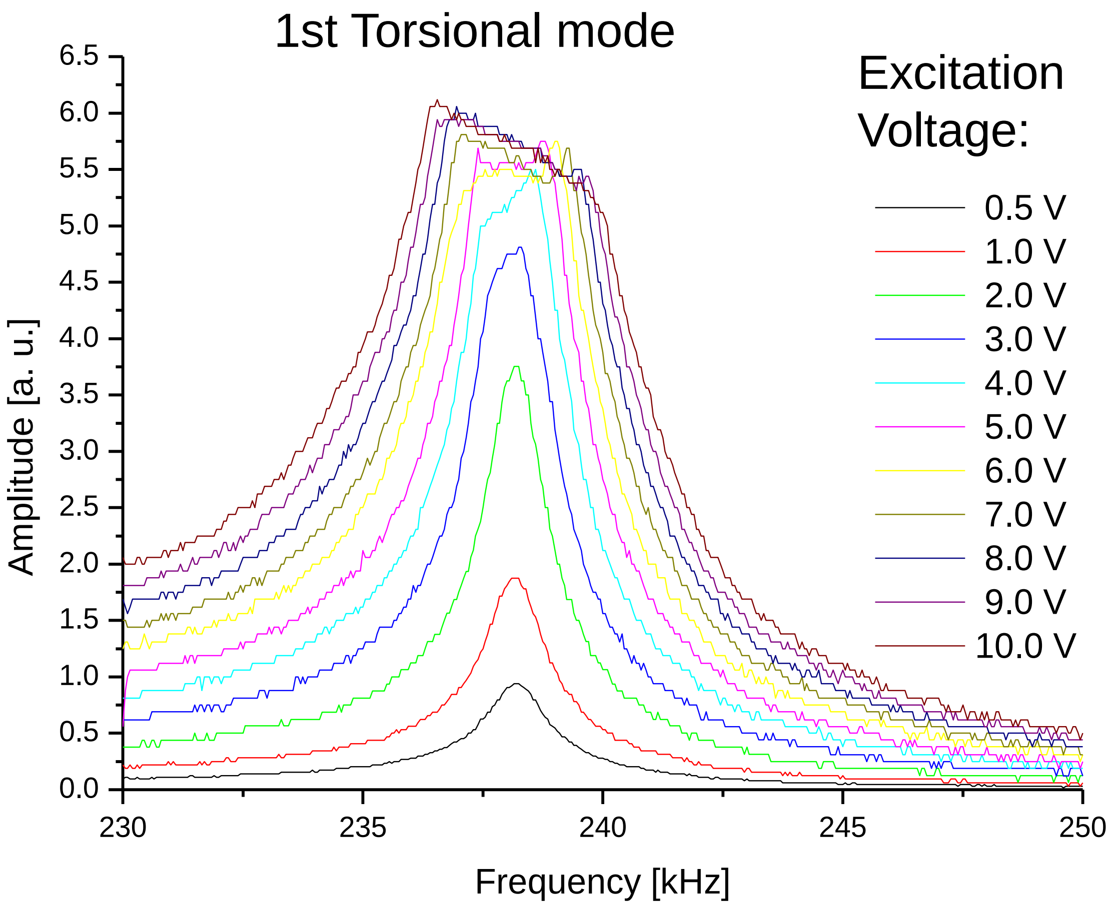
# AFAM contact resonance spectra, lateral

## Linear and nonlinear contact forces




Si-sample, Si-cantilever
$F_N$ =15 nN (static load)
$F_0$ =47 nN (pull-off force)
$k_c$ = 0.1 N/m
(cantilever spring constant)
$f_1$ = 200 kHz
$f_2$ = 600 kHz
(1st and 2nd free torsional resonance)

Lateral contact stiffness in the linear range of the tip-sample interactions:
$k^*_{lat} = 8aG^*$.
a = contact radius
G* = reduced shear modulus of the contact.



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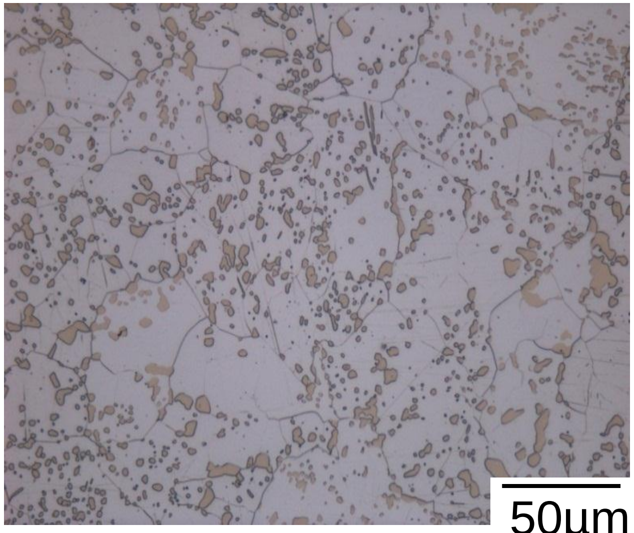
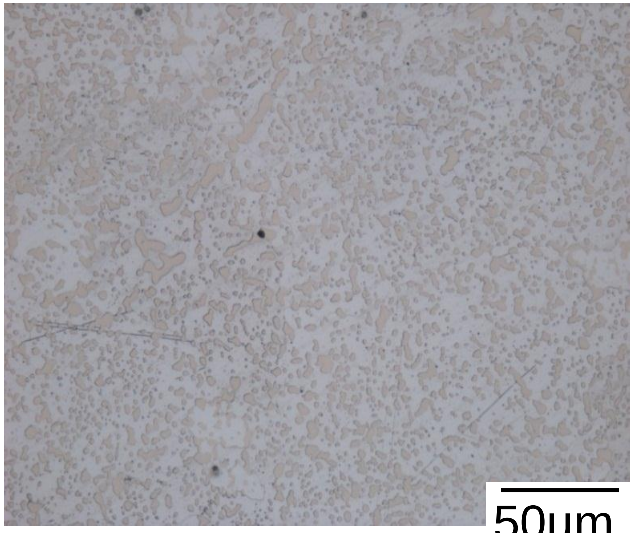
# Unalloyed steels: Fe-C systems (Fe - $Fe_3C$)

- Matrix: Ferrite ($\alpha$-Fe), cubic single crystal symmetry
- Precipitates: Cementite ($Fe_3C$), orthorhombic single crystal symmetry
- Macroscopic properties: dominated by the material properties as well as the size and shape of the $Fe_3C$-phase inclusions
- Large differences in the literature values of the material properties of cementite, probably differences between bulk and precipitate properties




Fe-0.8%C (C80)

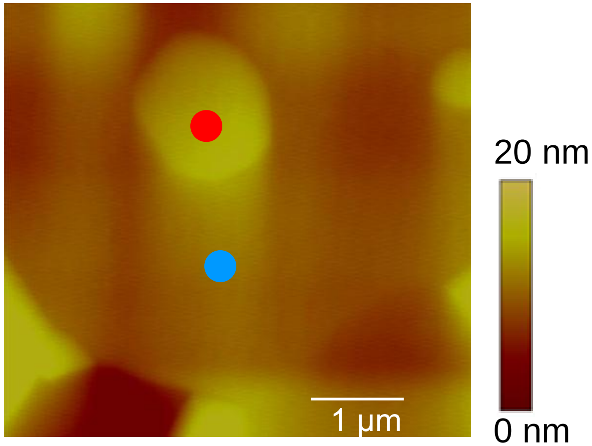



Fe-1.5%C (C150)

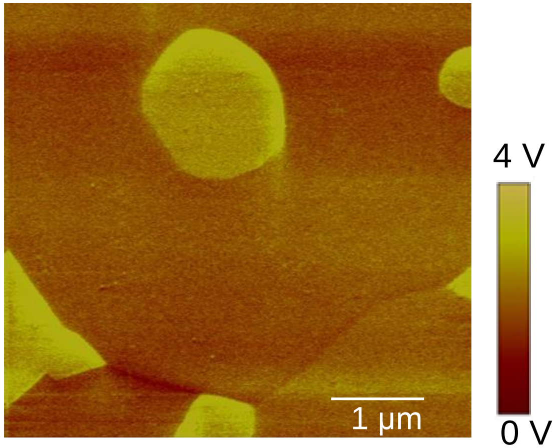
Microsections of two Fe-C-samples with a C-content of 0.8 and 1.5%, respectively, spherodized precipicitates

(Different contrast because of different edging procedures)

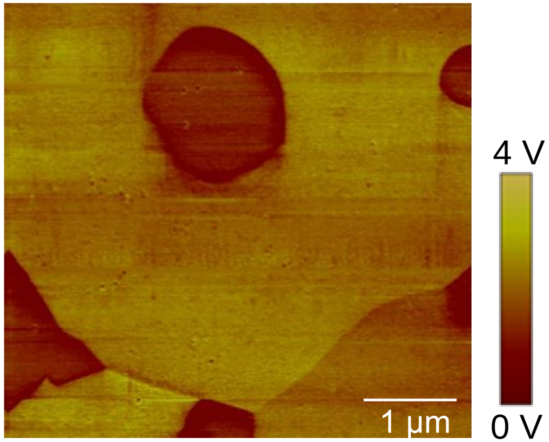
# Unalloyed steel (Fe-1.5%C), elastic stiffness mapping




AFM contact mode Topography

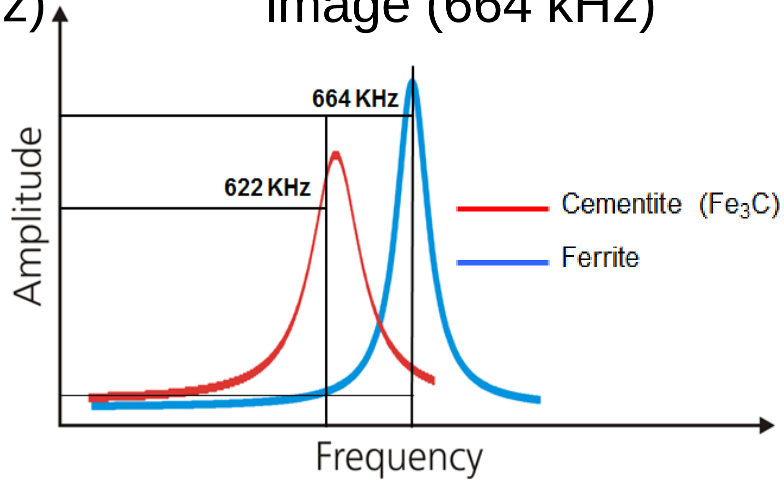



AFAM amplitude image (622 kHz)




AFAM amplitude image (664 kHz)

Frequencies close to the first contact bending resonance:
Higher contact resonances (i.e. higher stiffness) on ferrite ($\alpha$-Fe) than on cementite ($Fe_3C$);
Crystalline orientation??



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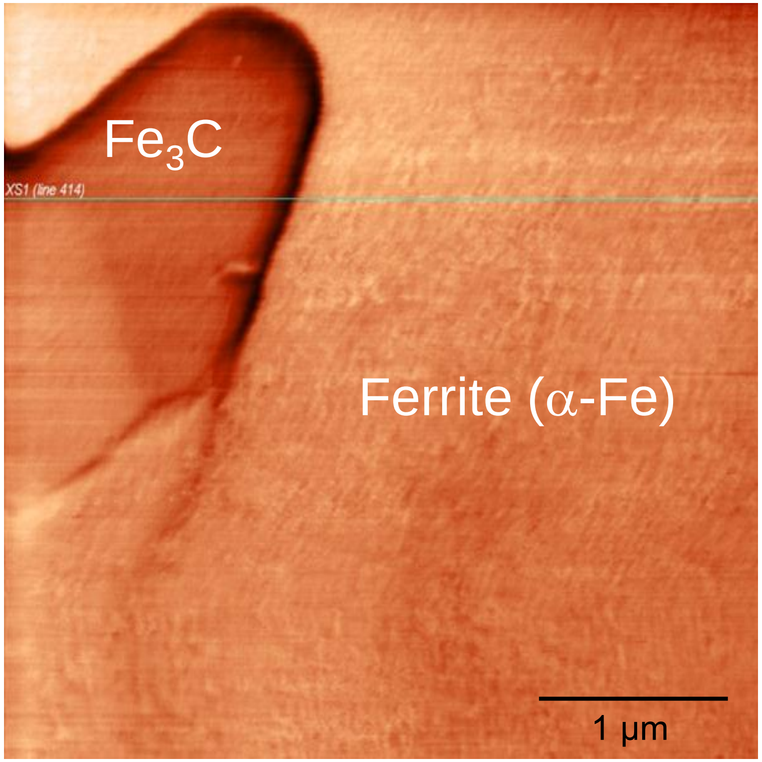
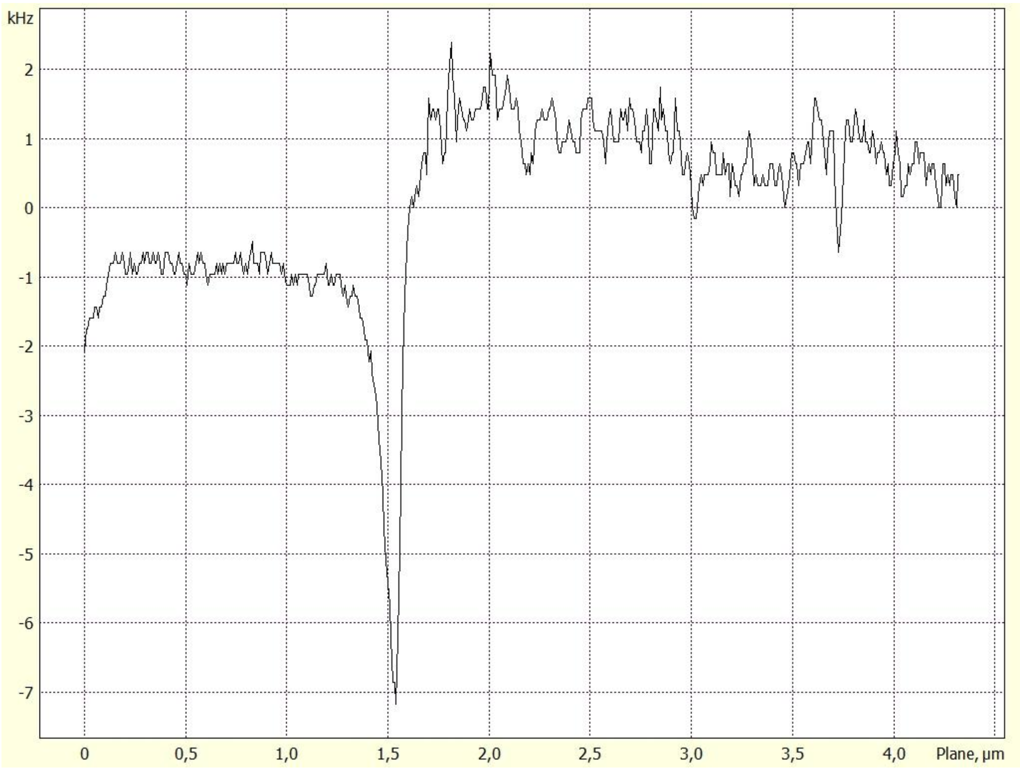
# Unalloyed steel (Fe-1.5%C), elastic stiffness mapping

AFAM (1st contact resonance)




Section analysis







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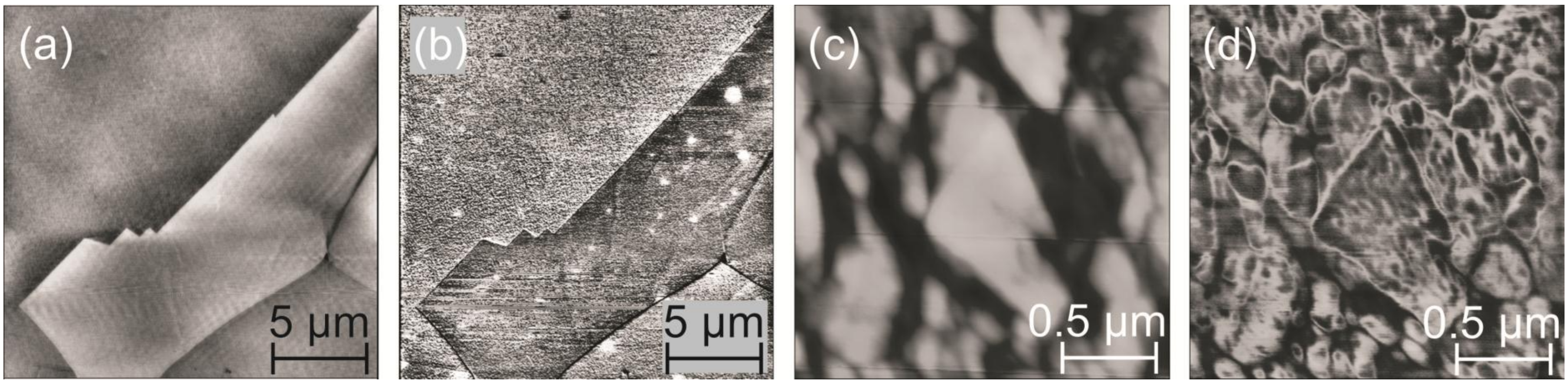
# AFM images of polycrystalline and nanocrystalline nickel

**Polycrystalline nickel**

**Nanocrystalline (nc) nickel grain size 30 nm**




Topography height scale 20 nm

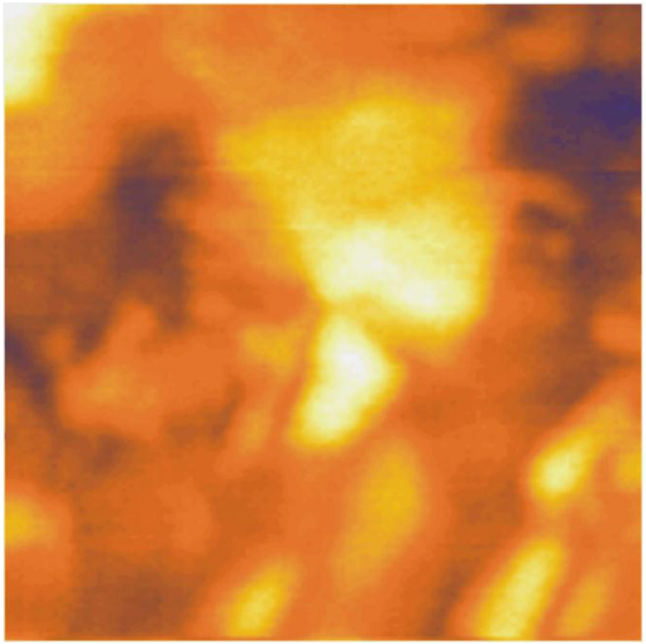
AFAM

Topography height scale 20 nm

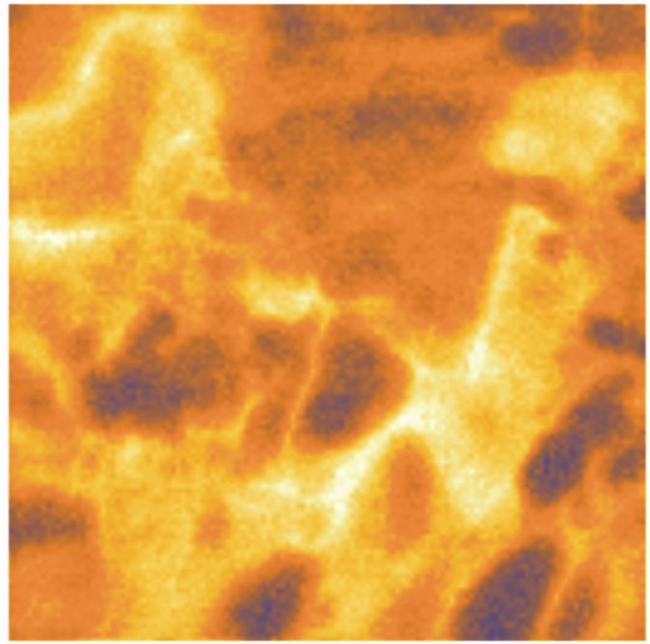
AFAM

AFAM: Amplitude images, first flexural contact resonance, frequency of operation close to 700 kHz

# Topography and AFAM image of nanocrystalline nickel



Topography image,
Height scale 10 nm
Image size: 1.5 x 1.5 $\mu m^2$



Contact resonance frequency AFAM image, frequency scale: 730 – 750 kHz

Cantilever spring constant: $k_c$ = 48 N/m,

Free bending resonances:
$f_1$ = 166 kHz
$f_2$ = 1031 kHz

Nanocrystalline nickel sample, grain size: 167 nm

U. Rabe, et al., Surface and Interface Analysis, Vol. 33 (2002) 65-70.



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# AFAM contact stiffness image of nanocrystalline nickel

Contact stiffness k* [N/m]

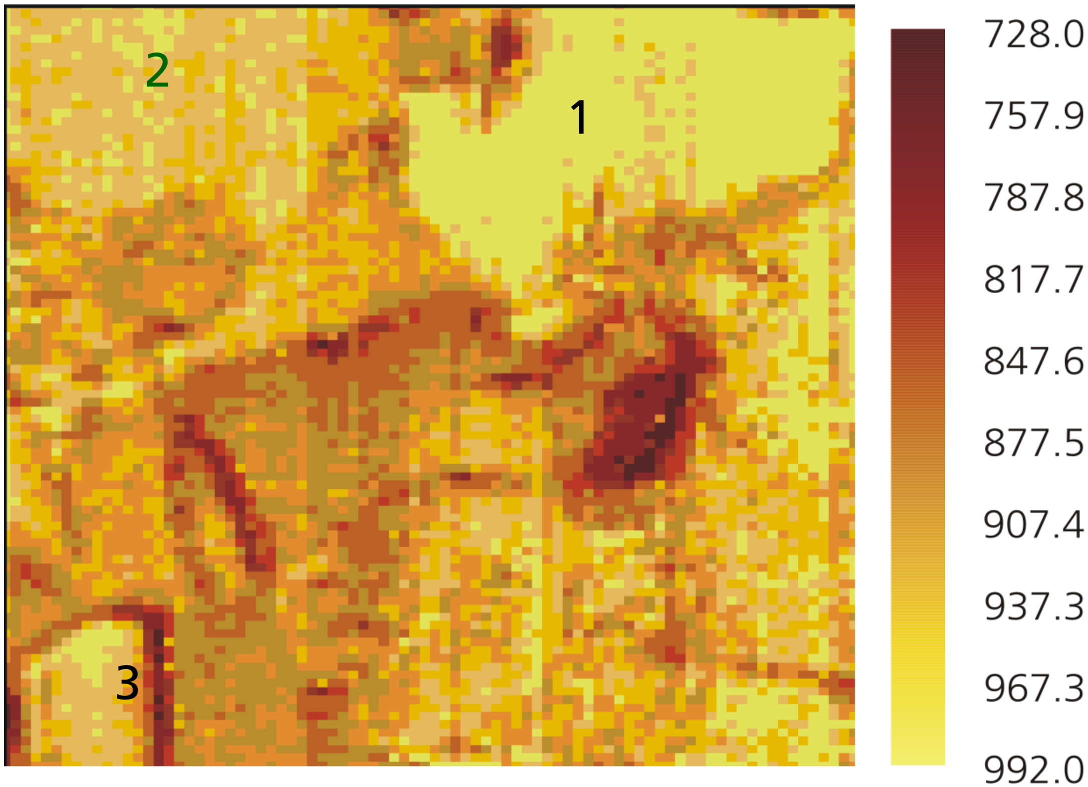


Image size: 1.4 x 1.5 µm²

$f_{\text{free modes}}$, $f_{\text{contact modes}}$ → Flexural Beam Theory → k*

Contact Stiffness k* → Hertzian Theory → Indentation Modulus M

Indentation modulus M of nickel:

Experiment [GPa]:

$M_{\text{Pos 1}} = 234$
$M_{\text{Pos 2}} = 184$
$M_{\text{Pos 3}} = 176$

Calculated from single crystal literature values [GPa]:

$M_{(111)} = 223$
$M_{(110)} = 218$
$M_{(100)} = 202$

U. Rabe, et al., Surface and Interface Analysis, Vol. 33 (2002) 65-70



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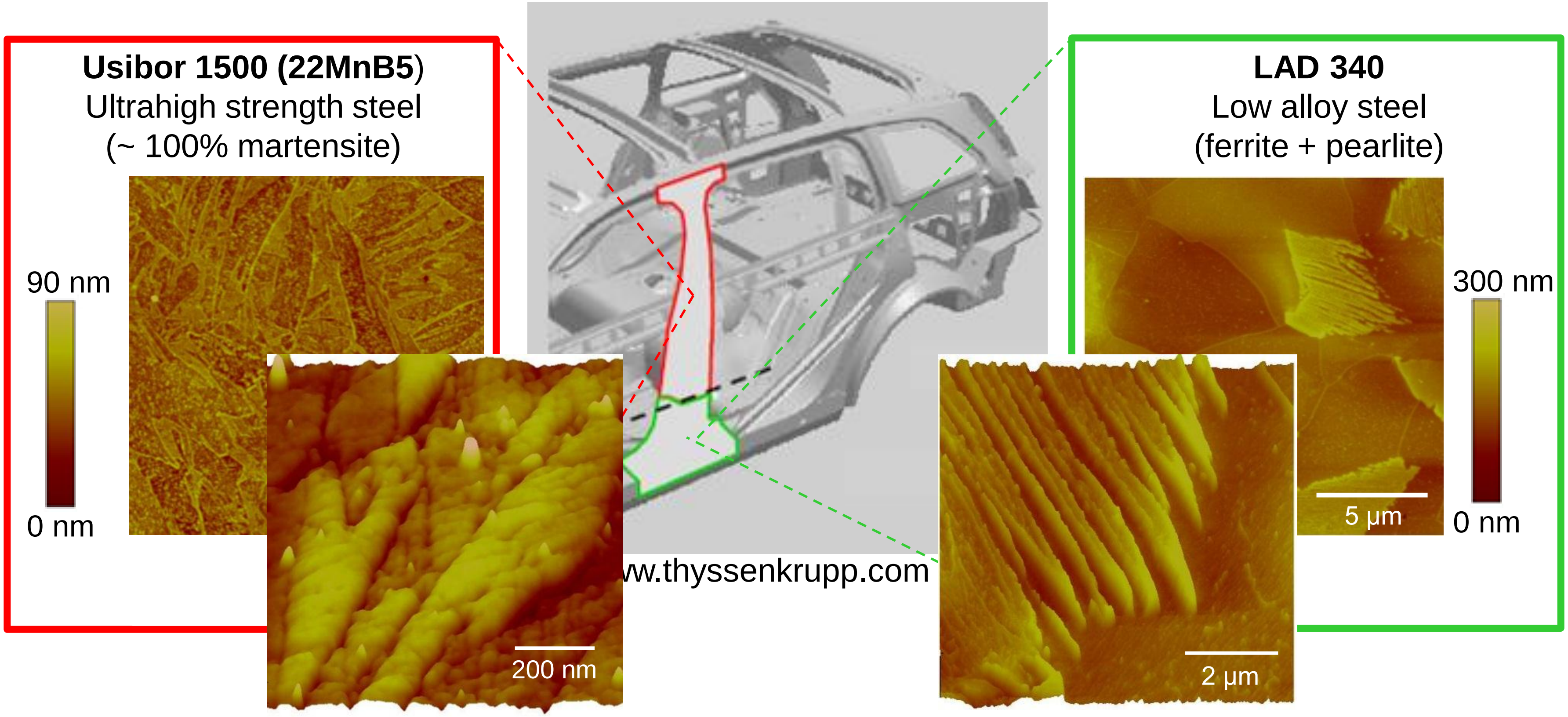
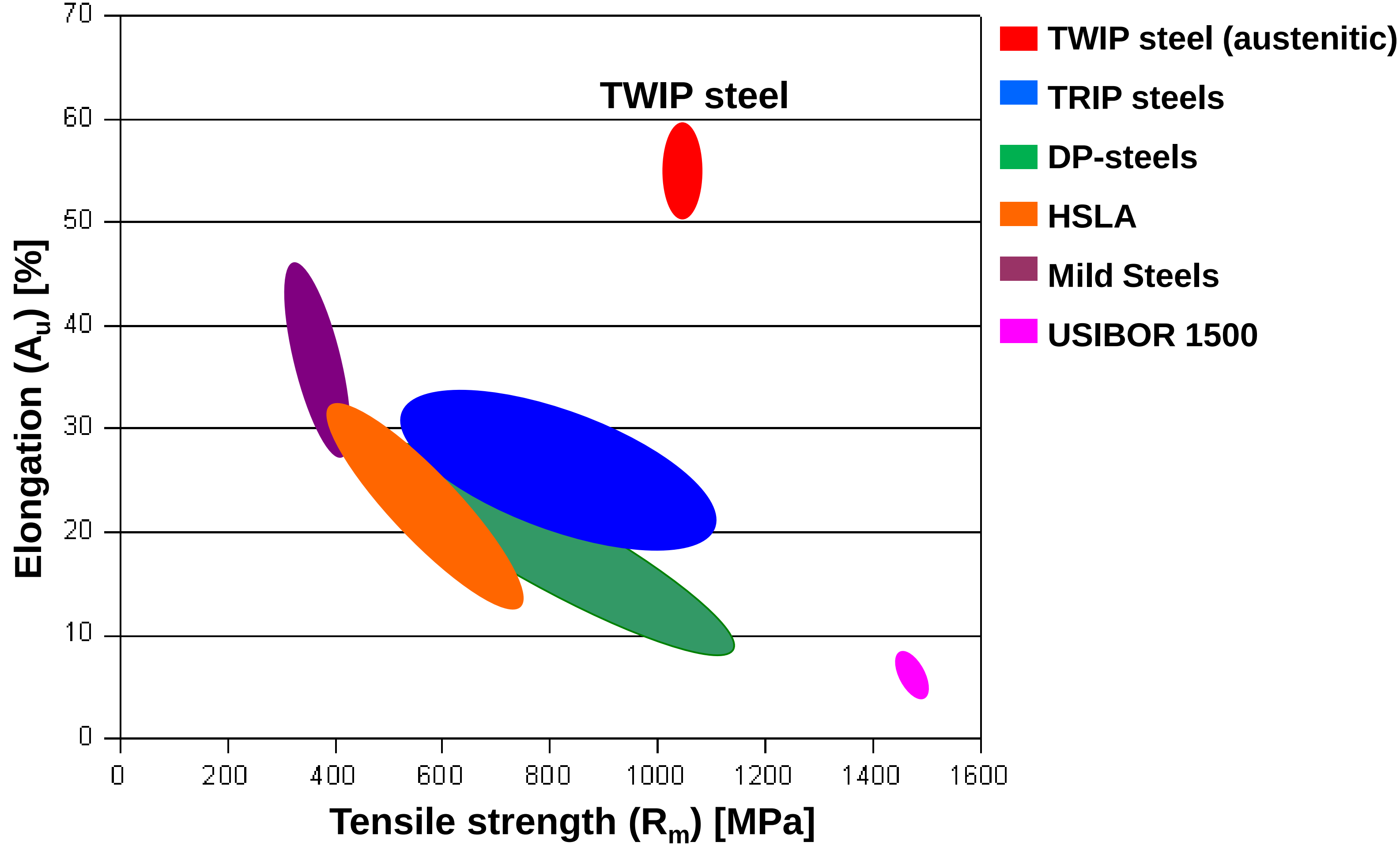
# Requirements to the crash behavior

- Maximum strength where crash deformation resistance is required;
- Good formability where high deformation takes place;
- Light weight steel design for low fuel consumption.



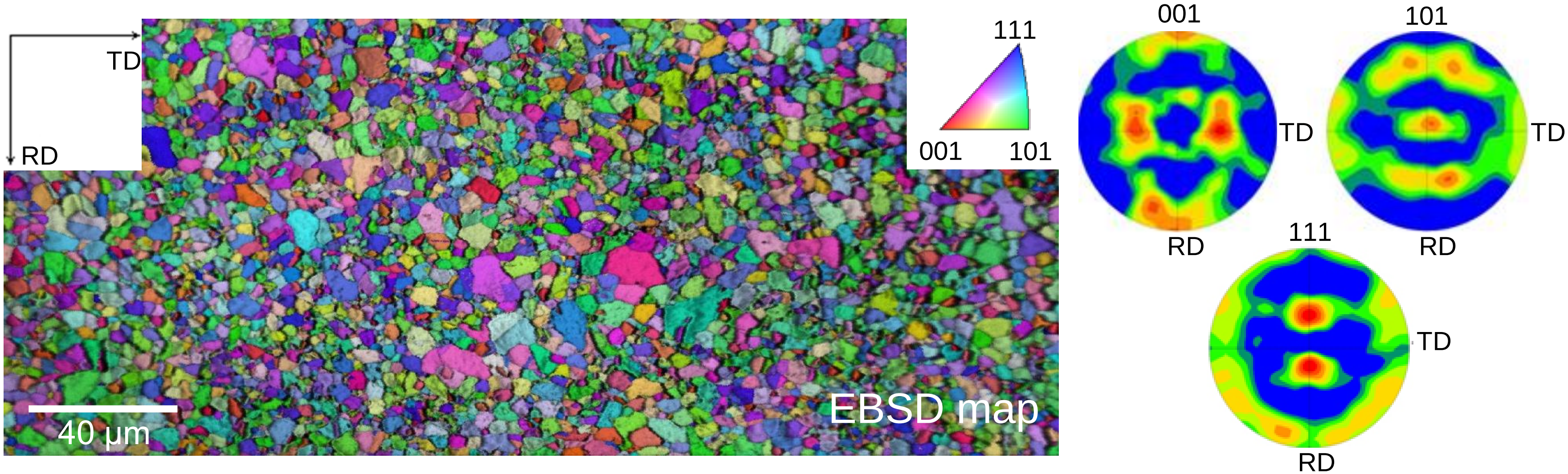
# Elongation and tensile strength of different steels




D. Cornette et al., *SAE Technical papers*: 2005-01-1327 (*modified*).

# TWIP steel, cold rolled and annealed

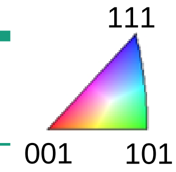
Chemical composition: Fe-22Mn-0.6C-0.2V (wt%);
Mechanical properties at room temperature: Yield strength ($R_{p0.2}$) > 600MPa, tensile strength ($R_m$) > 1000 MPa, uniform elongation ($A_u$) > 50%




Weak texture, elastic properties measured by ultrasonic methods:
Young's modulus: $E_{TD}$ = 186 GPa, $E_{RD}$ = 179 GPa;
Shear modulus: $G_{TD}$ = 72 GPa, $G_{RD}$ = 68 GPa;
Bulk modulus: $B_{TD}$ = 149 GPa, $B_{RD}$ = 162 GPa;
Poisson ratio: $\nu_{TD}$= 0.29, $\nu_{RD}$= 0.31.

RD: rolling direction
TD: transverse direction

# TWIP steel, EBSD and AFM topography image



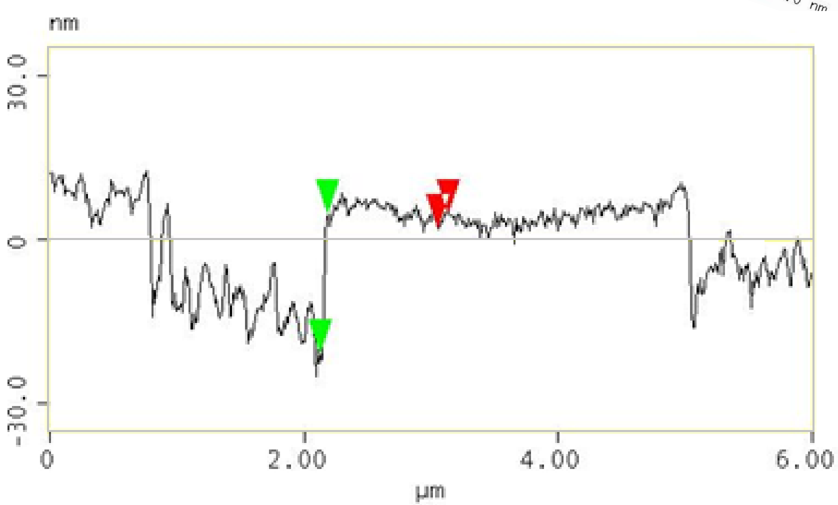

Height profile:

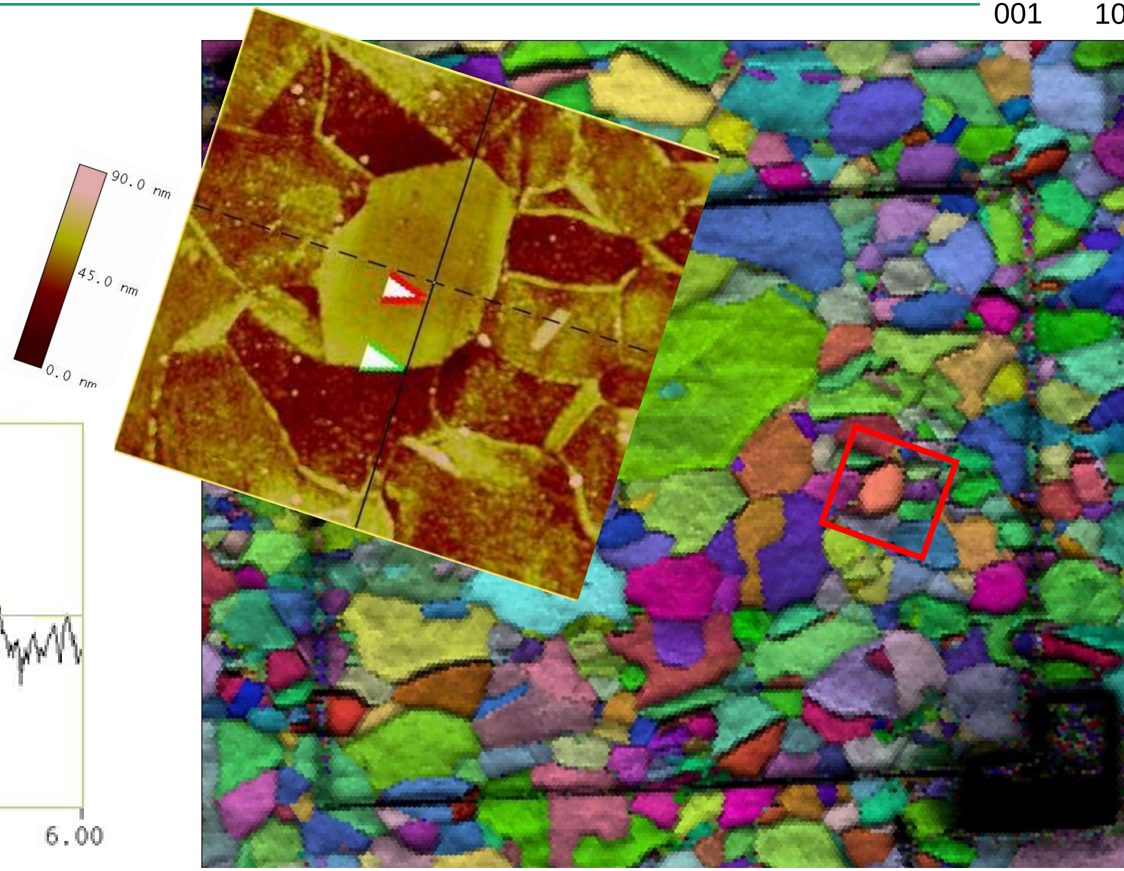
Horizontal distance: 82.031 nm

Vertical distance: 2.690 nm

Horizontal distance: 58.594 nm

Vertical distance: 25.752 nm




Austenite, average grain size 4 µm; EBSD map, AFM tapping-mode image; height profile along a line in the AFM image: on electrochemically polished surfaces largest height and lowest surface roughness for grains in/around [001] orientation



*S. Hirsekorn, U. Rabe, L. Batista, and L. Behl, 37th Annual Review of Progress in Quantitative Nondestructive Evaluation (QNDE), San Diego, USA, (2010).*

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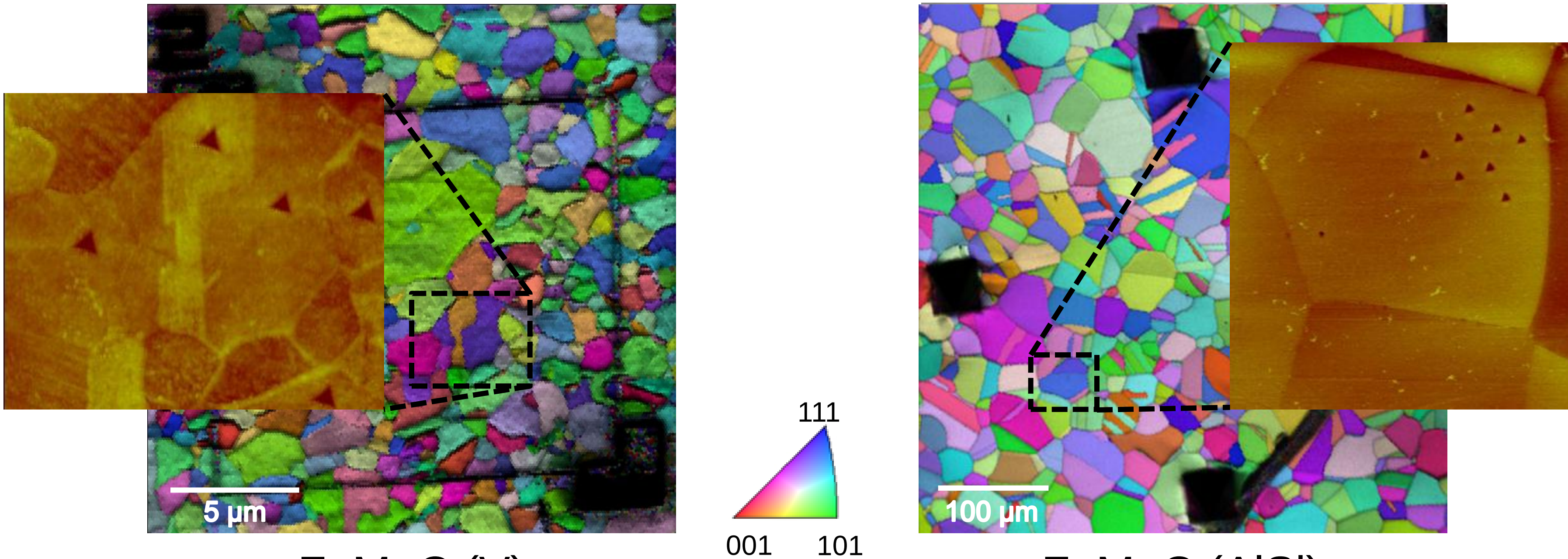
# TWIP steels – Indentation moduli [GPa]




FeMnC (V)    FeMnC (AlSi)

| | Nanoindentation | | | Quantitative AFAM | | |
|---|---|---|---|---|---|---|
| | (111) | (101) | (001) | (111) | (101) | (001) |
| FeMnC (V) | 218 | 207 | 197 | 179 | 167 | 139 |
| FeMnC (AlSi) | 160 | 134 | 131 | 171 | 142 | 113 |



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# TWIP steel: Tensile specimen preparation for in-situ AFM

Fe-22Mn-0.6C-0.2V (wt%), grain size 2 µm

Industrial steel plate (cold-rolled and annealed condition)

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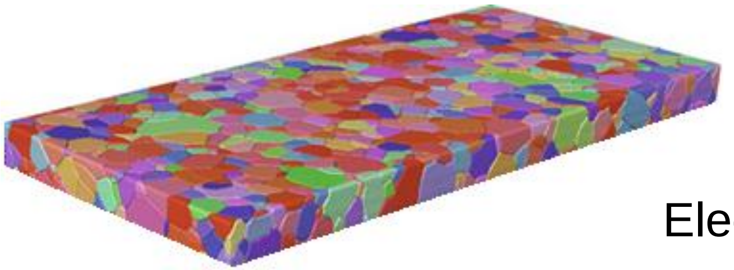
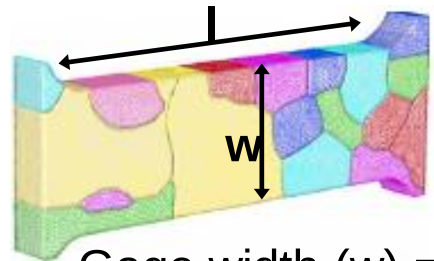


thickness (t) = 1.2 mm

Electroerosion cutting

Bone-shaped tensile sample




Gage width (w) = 0.5 cm
Gage length (l) = 1.5 cm

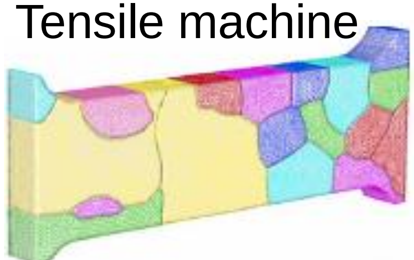
Mechanical and electrolytic polishing

Microindentation mark

Grain orientation determination (EBSD)

Tensile machine

σ



σ

Deformed in tension at room temperature to different strains

Atomic Force Microscope (AFM) observation

Objective: Observation of the formation of twins with increasing deformation



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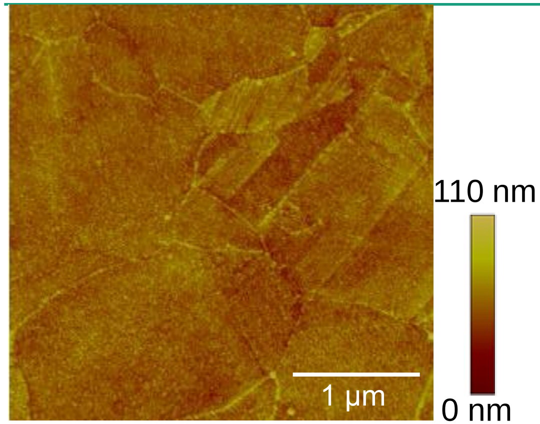
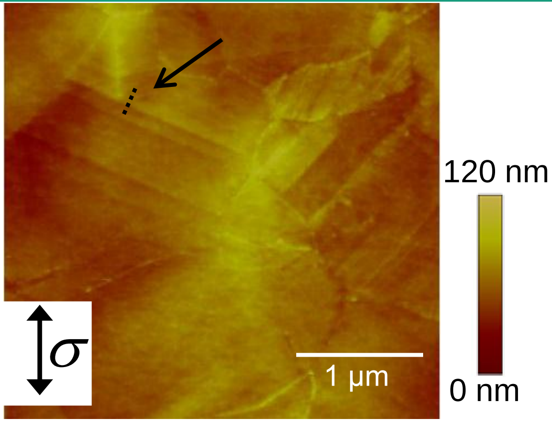
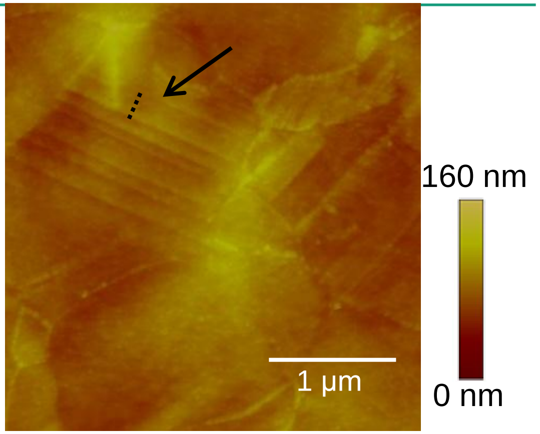
# Plastic deformation of TWIP steel, AFM and EBSD studies



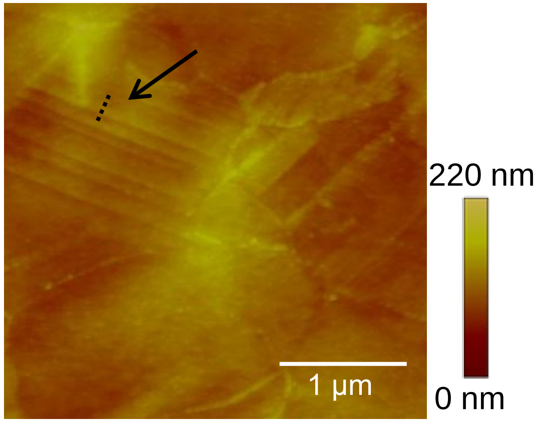

AFM tapping mode, topography
cold-rolled and annealed



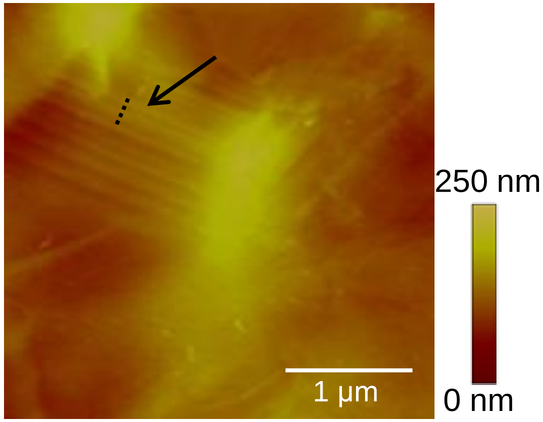

AFM contact mode, topography
3% plastic deformation



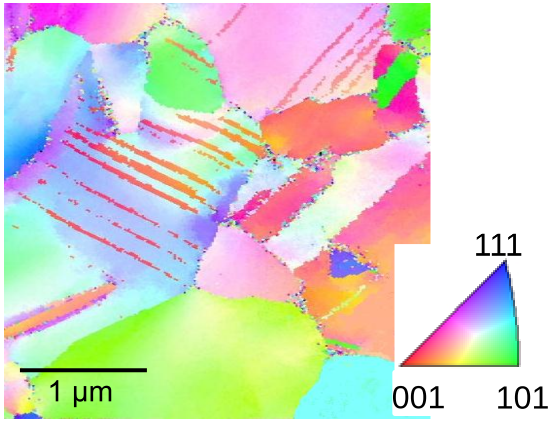

AFM contact mode, topography
6% plastic deformation




AFM contact mode, topography
10% plastic deformation




AFM contact mode, topography
15% plastic deformation




EBSD map
15% plastic deformation



Formation of twins with increasing deformation

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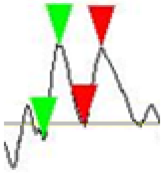
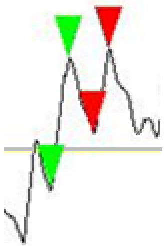
# Plastic deformation of TWIP steel, AFM section analysis

## Topography

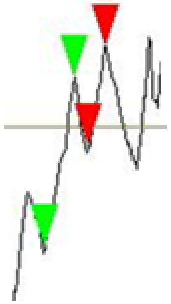
3% plastic deformation
Vertical distance: 10.889 nm
Vertical distance: 8.409 nm



6% plastic deformation
Vertical distance: 13.667 nm
Vertical distance: 16.336 nm



10% plastic deformation
Vertical distance: 14.567 nm
Vertical distance: 22.338 nm



15% plastic deformation
Vertical distance: 17.393 nm
Vertical distance: 29.541 nm

Strong influence of grain orientation on deformation twinning:

- For low strain: Grains with <111> orientation parallel or close to the tensile axis were twinning deformed first.
- For larger deformation: Grains with unfavorable <001> orientation parallel or close to the tensile axis were twinning deformed also.

# Unalloyed steels: Fe-C systems (Fe - $Fe_3C$)

Matrix: Ferrite ($\alpha$-Fe); Precipitates: Cementite ($Fe_3C$)

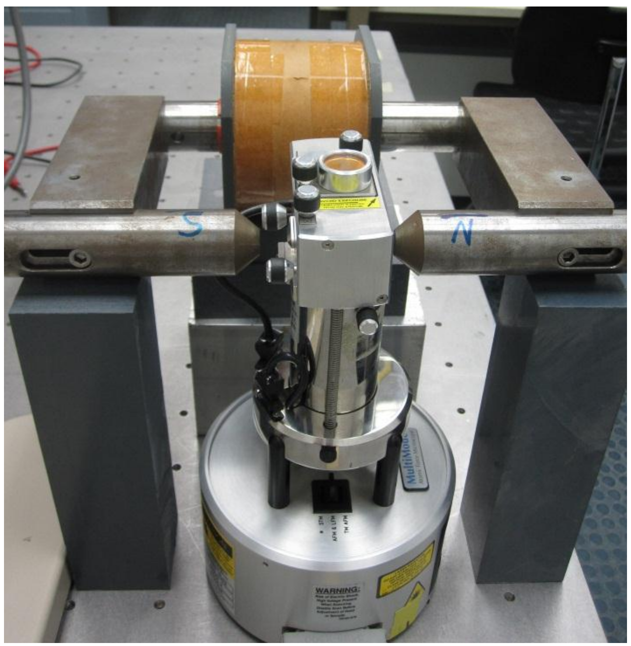
- Macroscopic properties: dominated by the material properties as well as the size and shape of the $Fe_3C$-phase inclusions
- Interdependence between mechanic and magnetic properties
- Interactions of magnetic domains with the microstructure influence the steel properties

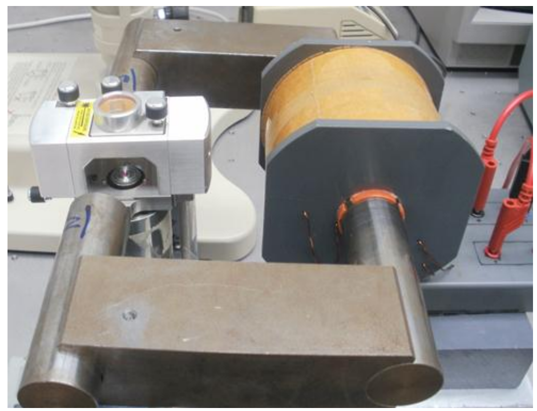
| | **α-Fe** | **$Fe_3C$** |
|---|---|---|
| Crystalline structure | body-centered cubic (BCC) | orthorhombic |
| Mechanical properties | soft & ductile | hard & brittle |
| Magnetic properties (MD: magnetic direction) | $T_C \approx 766°C$ | $T_C \approx 215°C$ |
| | $E_{MD}$: [100], [010], [001] | $E_{MD}$: [001] |
| | $H_{MD}$: [111] | $H_{MD}$: [010] |

$E_{MD}$: easy direction, $H_{MD}$: hard direction; $T<T_C$: ferromagnetic, $T>T_C$: paramagnetic

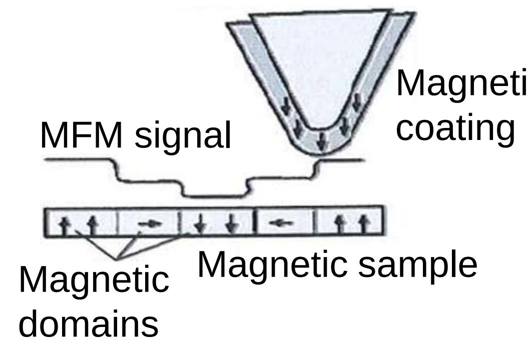
# Magnetic Force Microscope coupled with an external coil

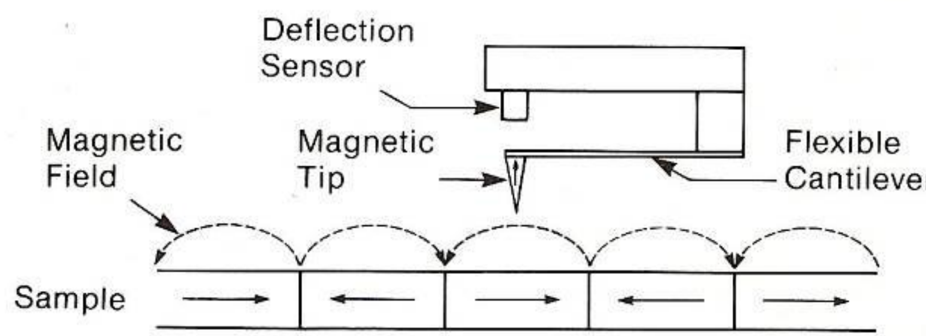
A magnetic force microscope (MFM) maps magnetic domain structures at a surface of magnetic samples by recording the force gradient between a sample and a tip coated with a ferromagnetic material; the external coil provides with a controlled in-plane  magnetic field.





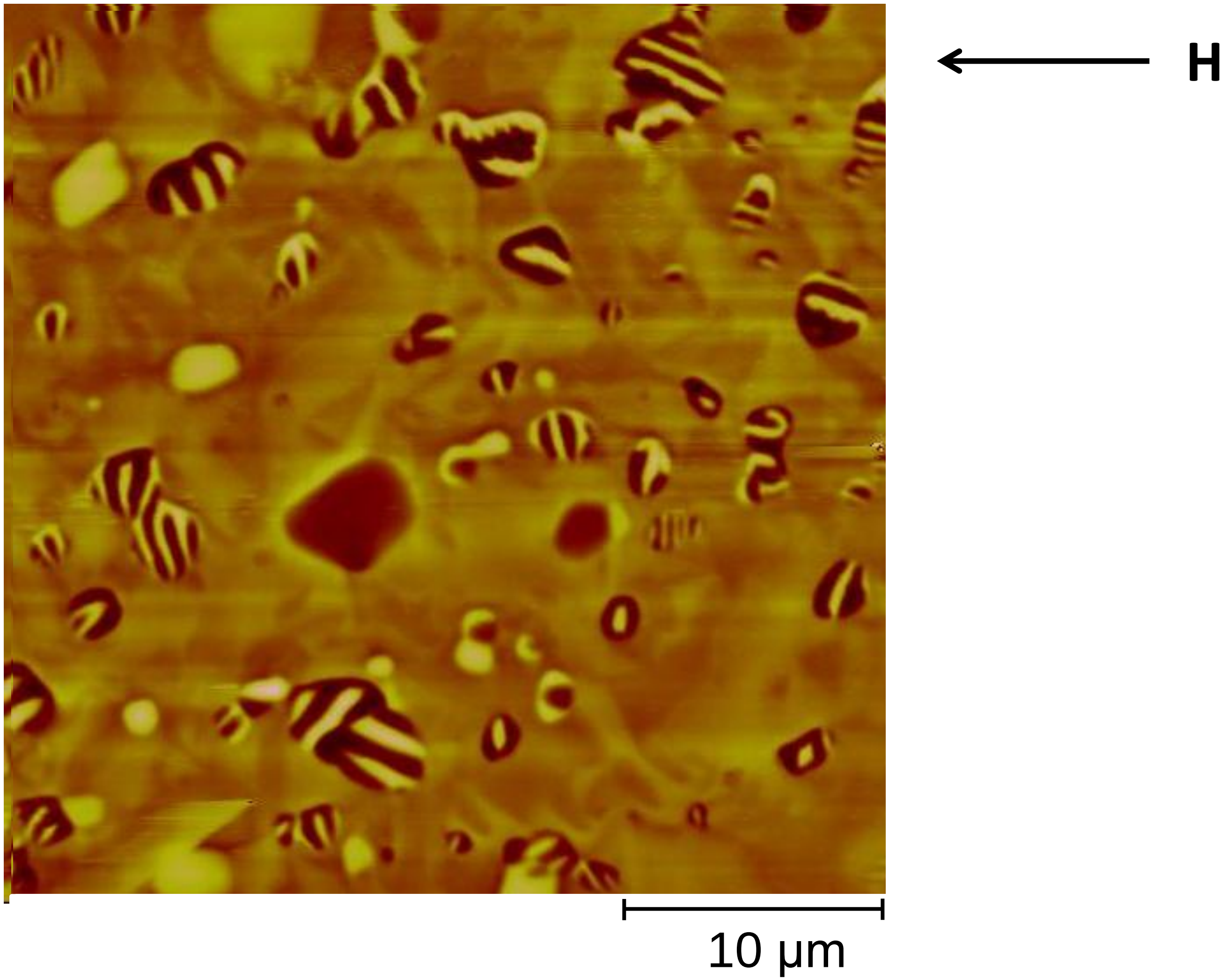
Magnetic Force Microscope



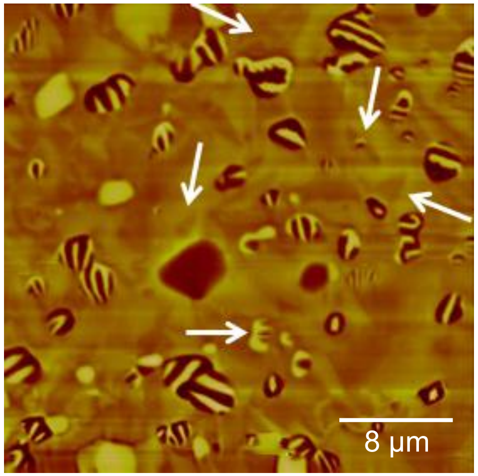




P. Grütter et al. in*: Scanning Tunneling Microscopy II, (*1992).

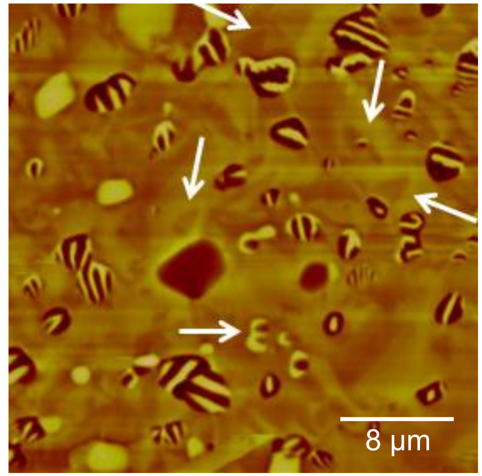
# Magnetic domain dynamics in $Fe\text{-}Fe_3C$ (Fe-1.5%C)



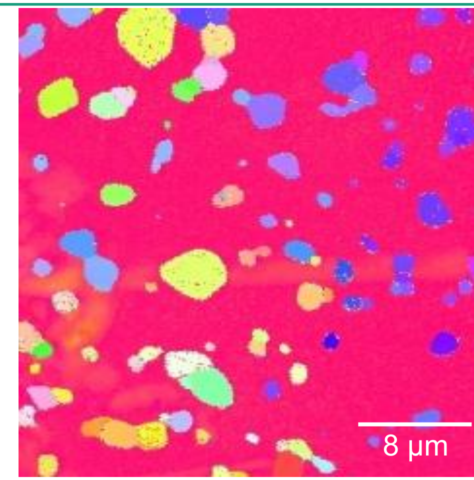
# Magnetic microstructure in Fe-1.5%C, MFM and EBSD studies



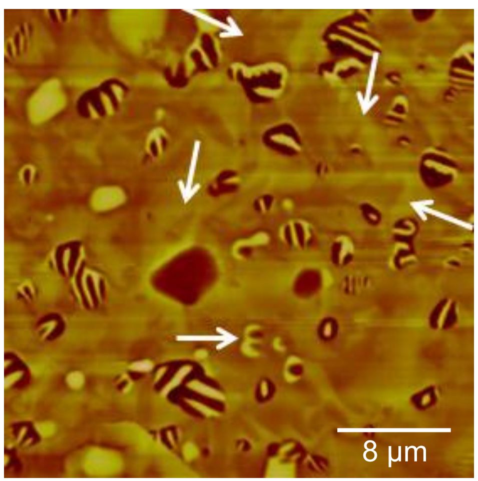

MFM (H=0)



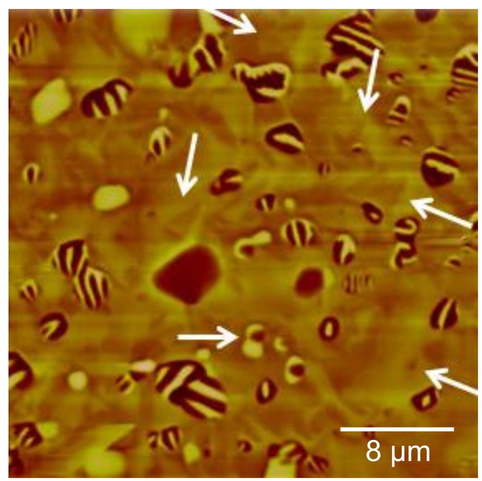

H = 13 mT



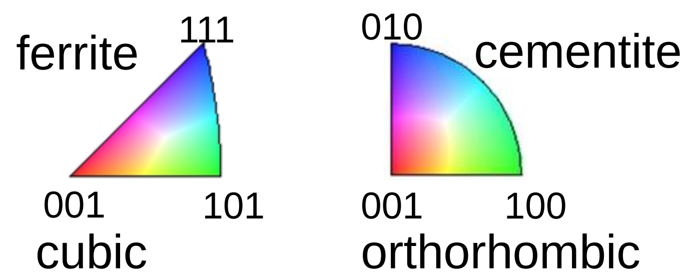

EBSD map




H = 20 mT




H = 24 mT




← H



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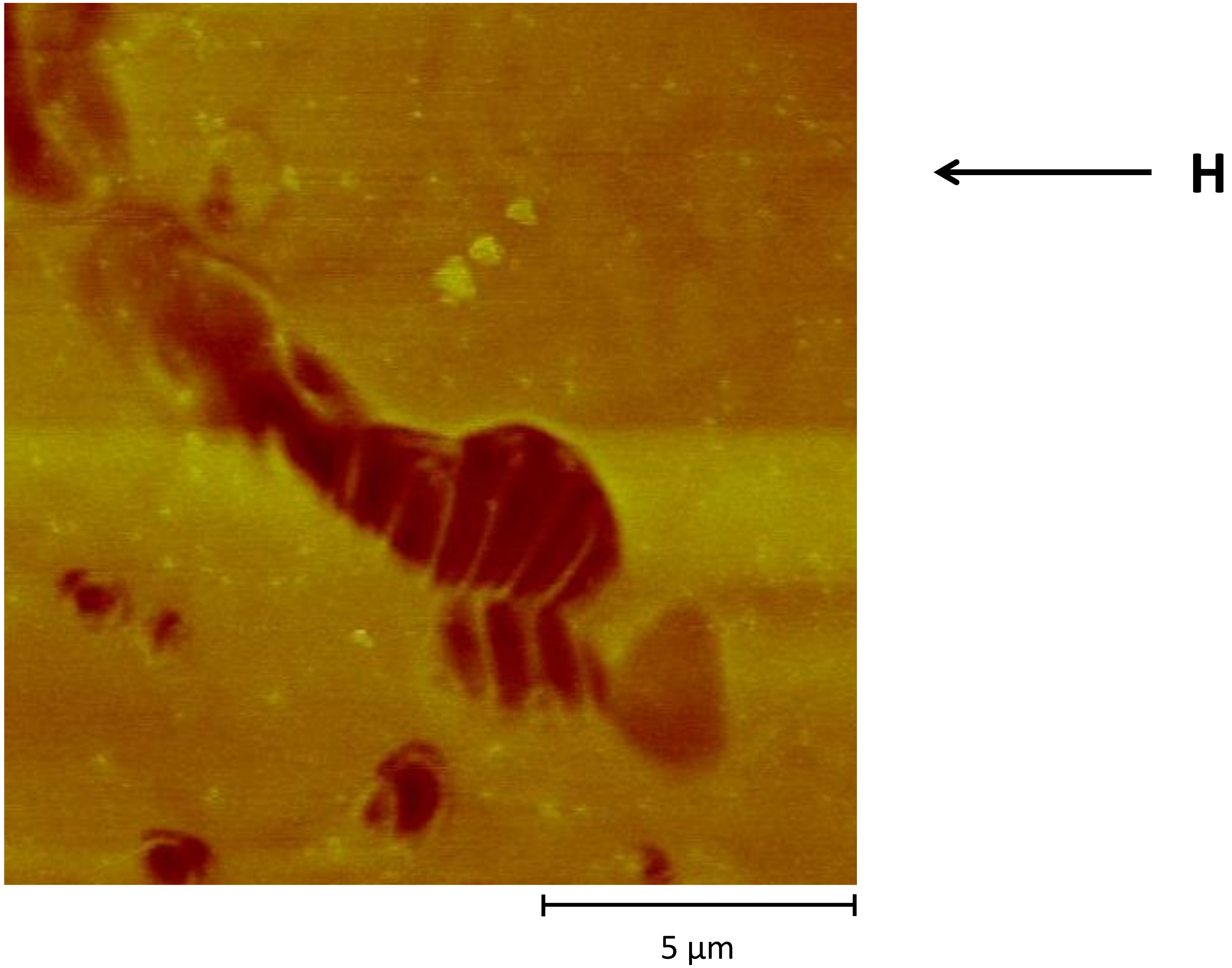
# Magnetic domain dynamics in cementite ($Fe_3C$)



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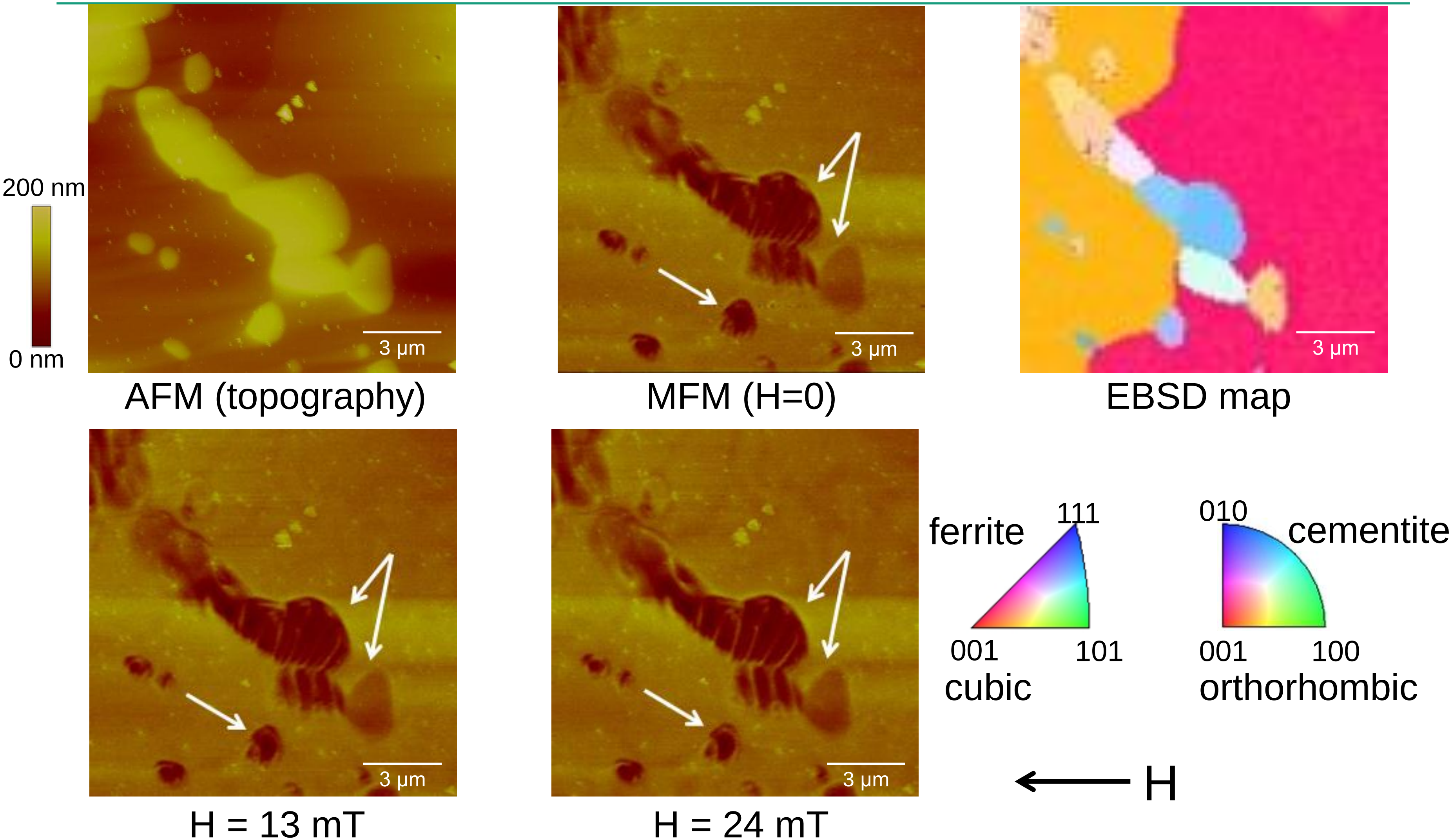
# Magnetic microstructure in $Fe_3C$, MFM and EBSD studies



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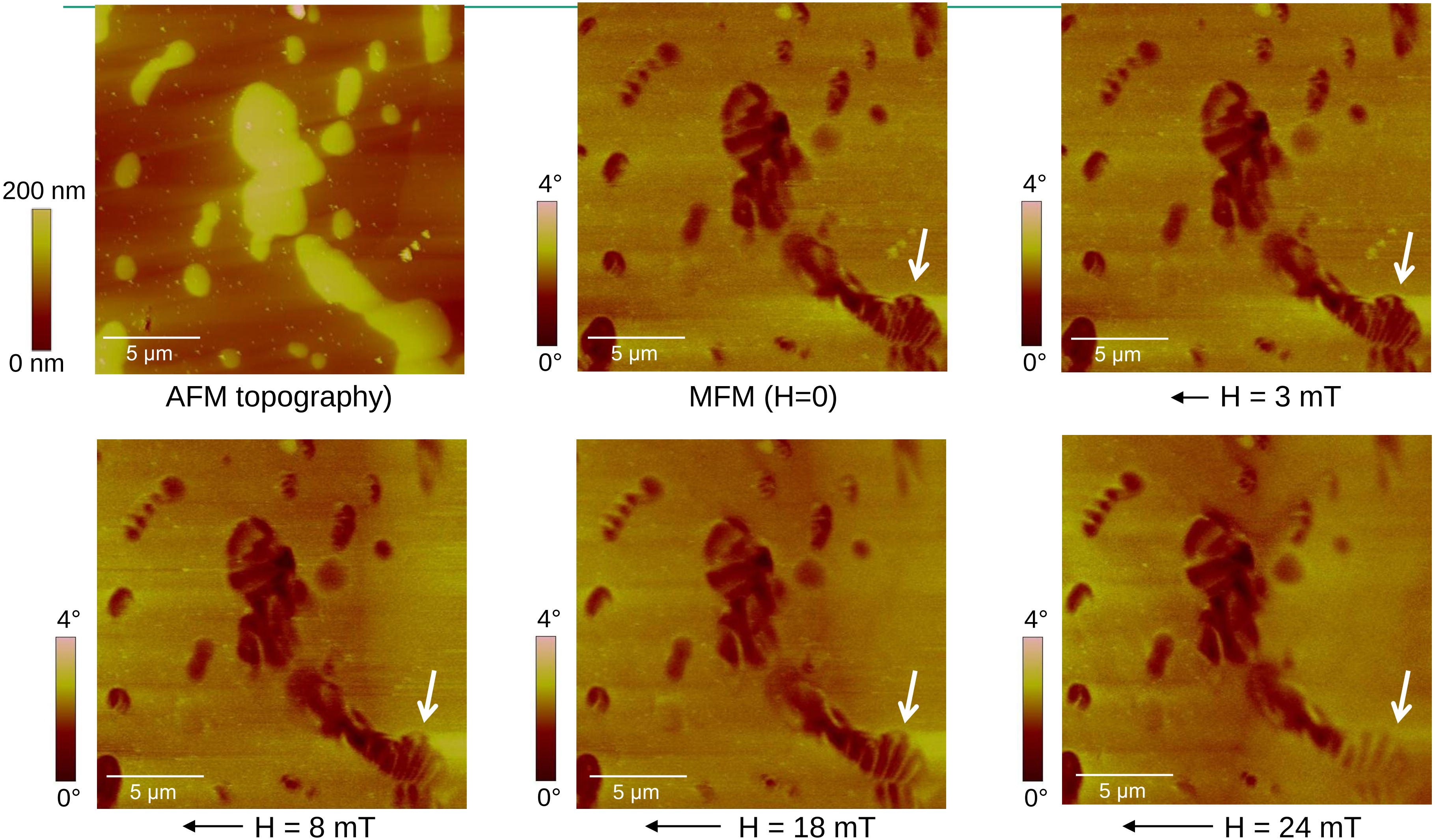
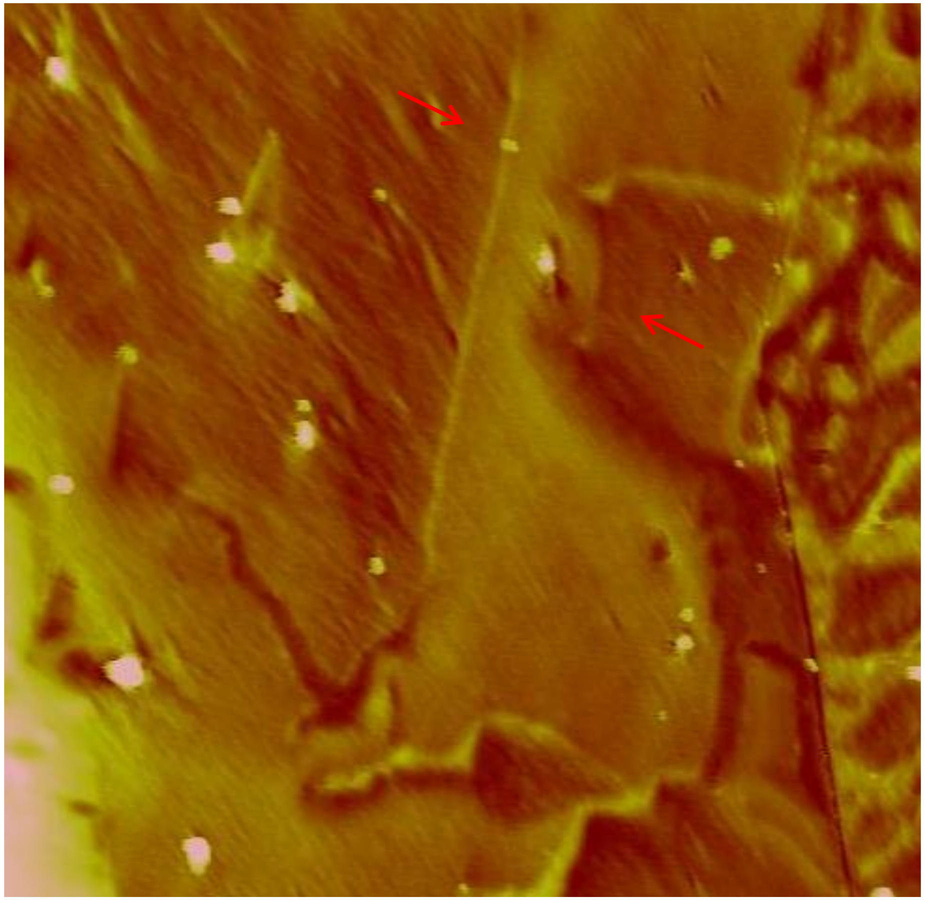
# MFM - magnetic microstructure of $Fe_3C$



# Domain wall dynamics in soft iron



10 µm

**H = 0**

# Summary

- Motivation for AFM in ndt and quality assurance: materials design by selective adjustment of microstructures controling macroscopic properties (adaptation to special application cases)
- Principle of AFM and its different operation modes
  - quasi-static, non-resonant dynamic, resonant dynamic
  - contact, intermediate-contact, non-contact (force contact), intermittent contact
  - different interaction forces
  - access to different local material properties
- Applications to metals
  - nanocrystalline nickel
  - unalloyed steel
  - TWIP steel

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