Fatigue of nanostructured materials

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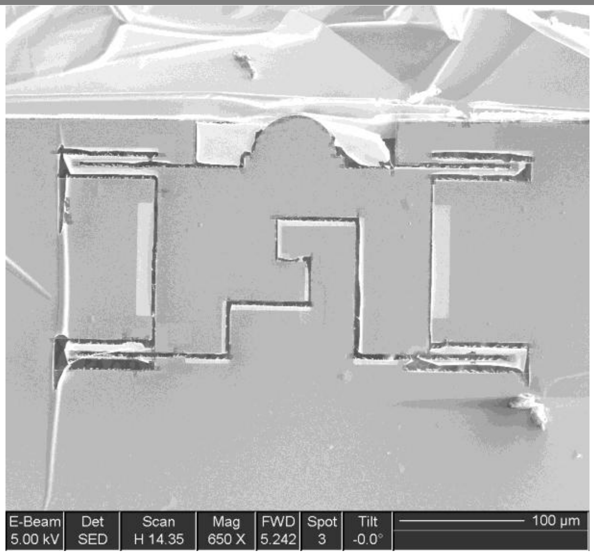
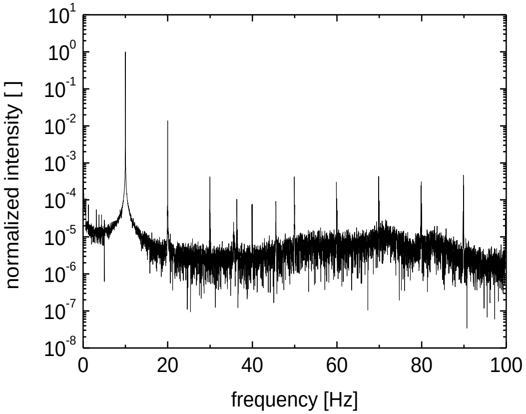
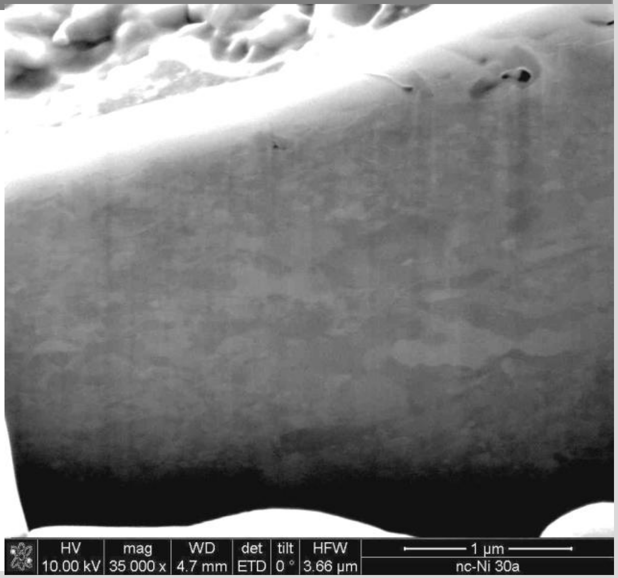
Institute of Applied Materials, KIT





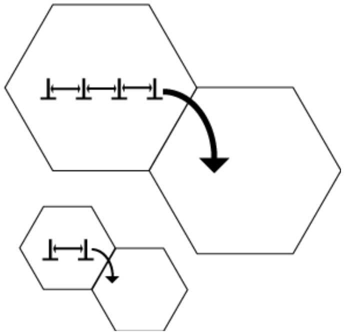


Overview - Projects

- Fatigue of nc-Ni: investigation of deformation mechanisms
- *In-situ* tests on nc-Cu: to observe microstructure during fatigue
  (with A. Castrup, INT, A. Minor, LBNL, Berkeley, USA)
- Nanotwinned (nt)-Cu: determination of tensile and fatigue properties
  (with T. Kennerknecht, KIT, Xingjang Chang, Texas, USA)
- **F**ourier **T**ransform (FT) analysis of fatigue data: tool to quantify nonlinearity and inelasticity
  (with V. Baroso, J. Höpfner, M. Wilhelm, Polymer Chemistry, KIT)

Grain size > 100 nm

Grain size < 100 nm



$$\sigma = \sigma_0 + \frac{k_y}{\sqrt{D}}$$

Properties:
• Enhanced properties following the Hall-Petch relation (strength, hardness, wear resistance)
• Enhanced fatigue properties in HCF
• Reduced ductility due to reduced dislocation activity

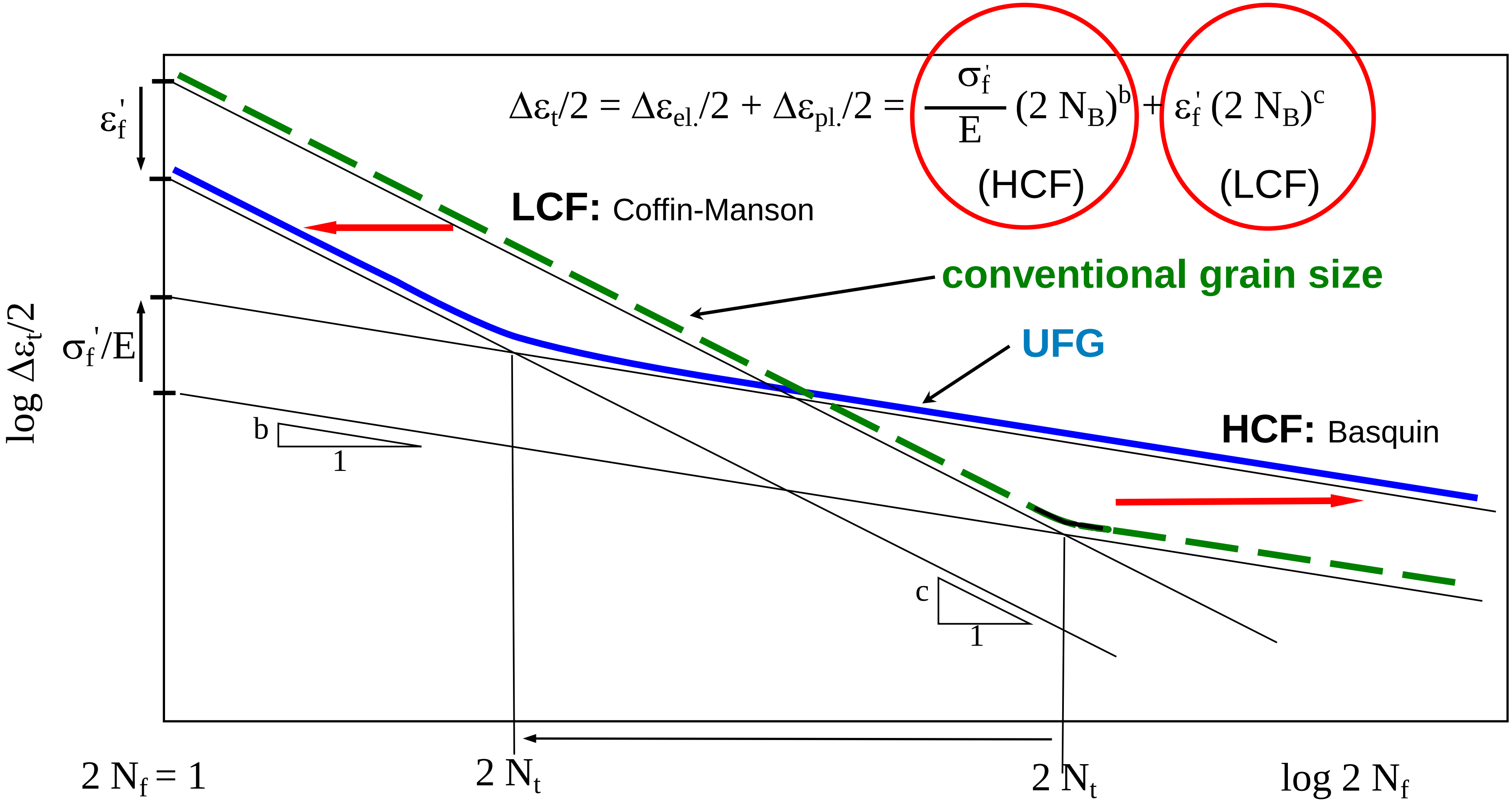
[Padilla & Boyce, ***Exp Mech,*** 2006]
[Kumar et al., ***Acta Mater,*** 2003]

Interests:
➢ Different deformation mechanisms as in cg materials
➢ Size / scaling effects
➢ Grain coarsening ↔ crack initiation

[Furuya et al., ***Scr. Mat.***, 2008]
[Simons et al., ***Mater Sci Eng A***, 2006]

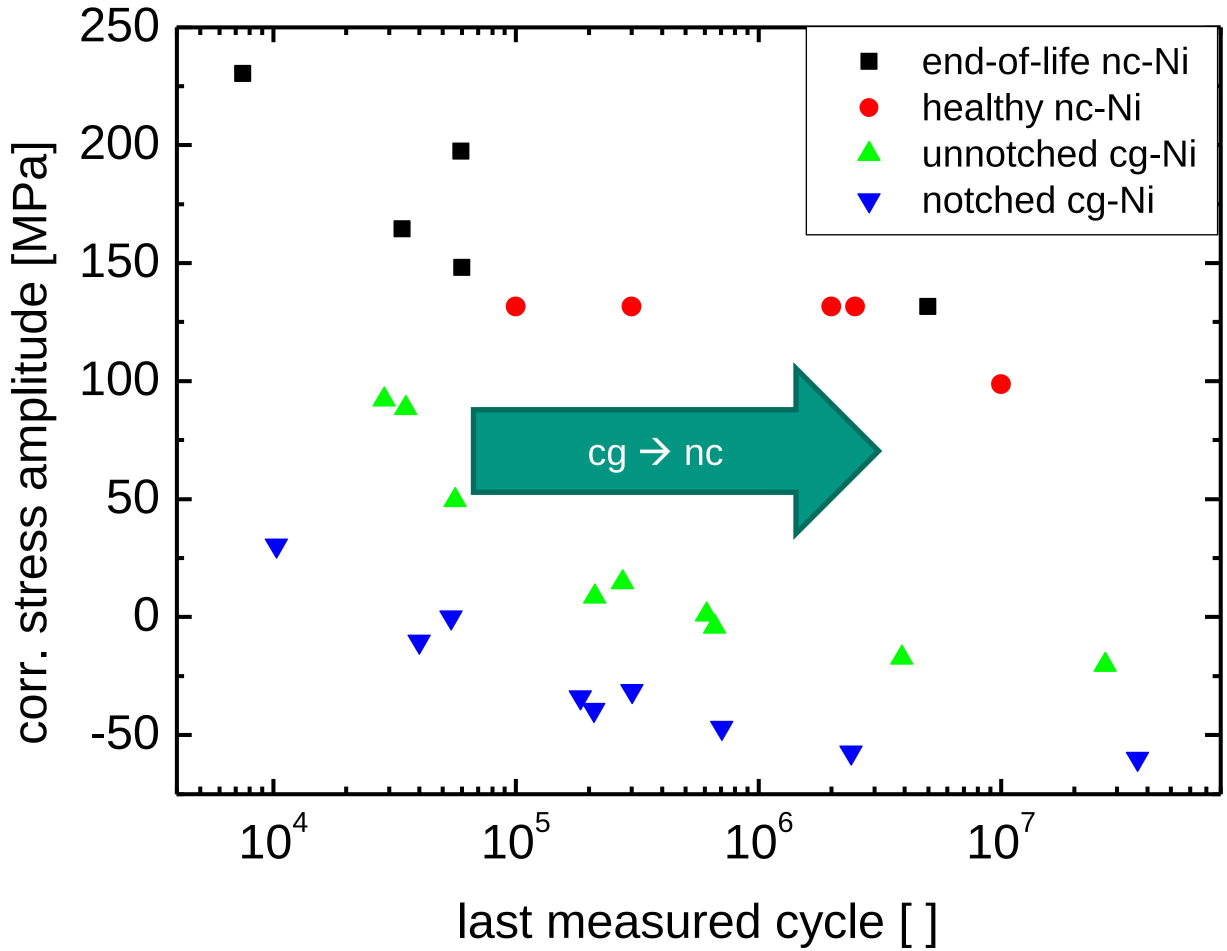
fatigue of nanocrystalline materials

Fatigue of nc materials



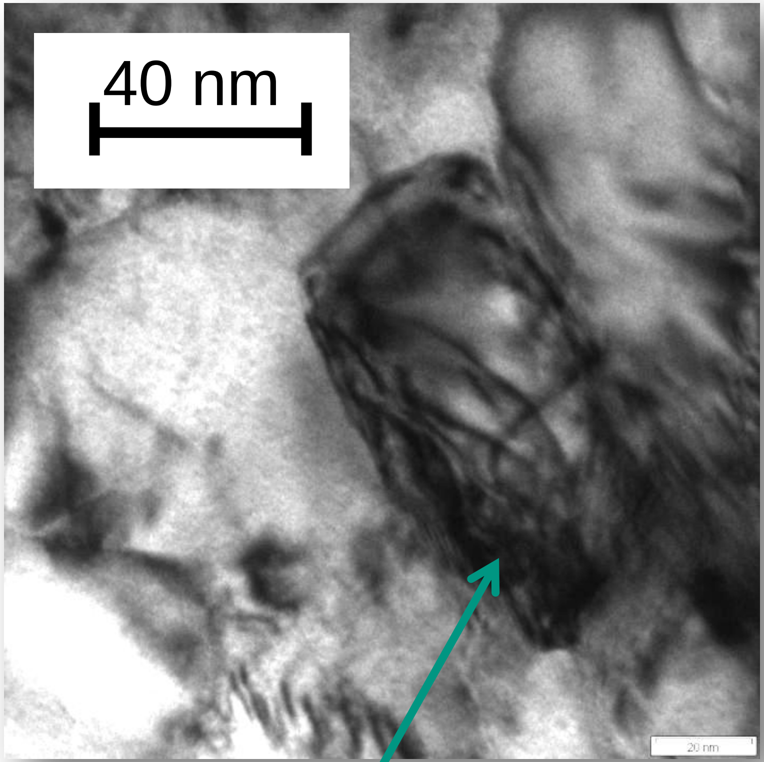
[Mughrabi & Höppel, ***MRS***, 2000]

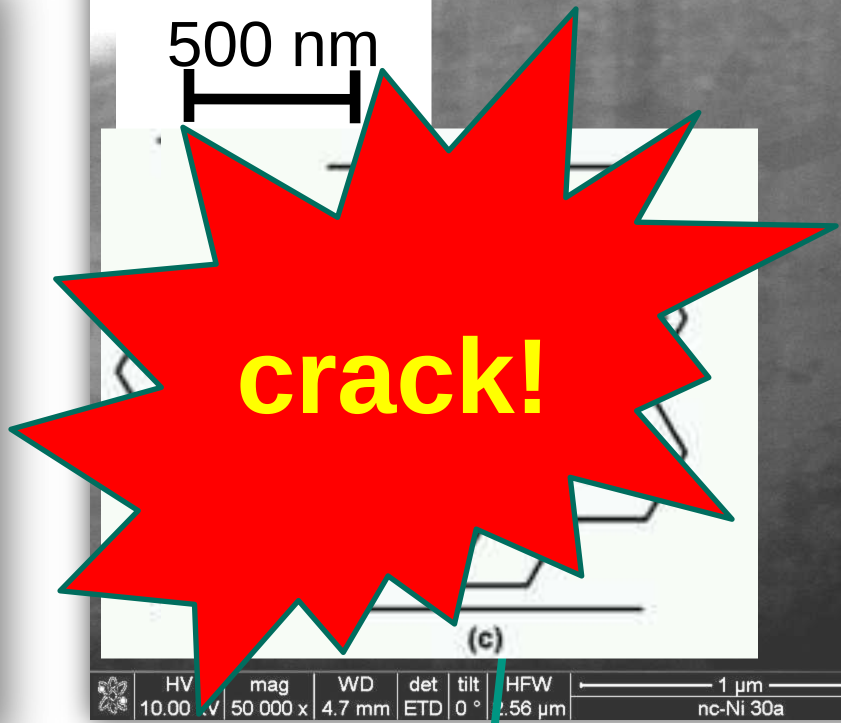
Fatigue of nc materials



σ_m (cg-Ni)= 200 MPa values from [Son, et al., ***Scripta Mater 50,*** 2004]
σ_m (nc-Ni)= 200 – 300 MPa

Fatigue of nanocrystalline metals - Motivation

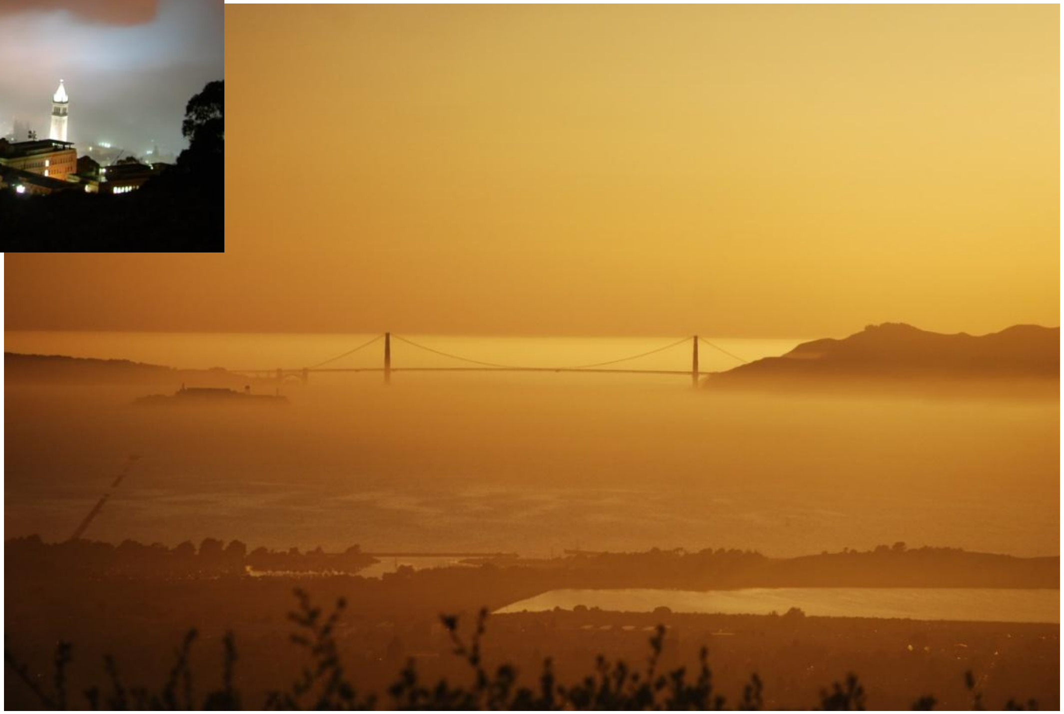




What happens inside the „nano-grains“? Full dislocations or only partials and twins?

Grain rotation could lead to coalescence (grain coarsening).

[Meyers, ***Progr Mater Sci,*** 2006]



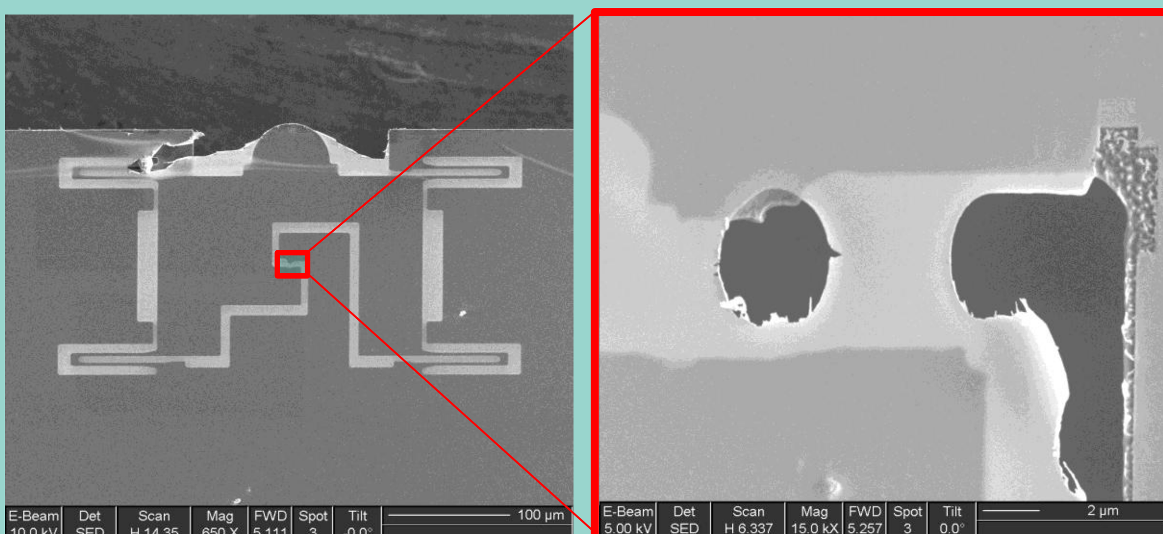
In-situ fatigue of nc-Cu

In-situ TEM

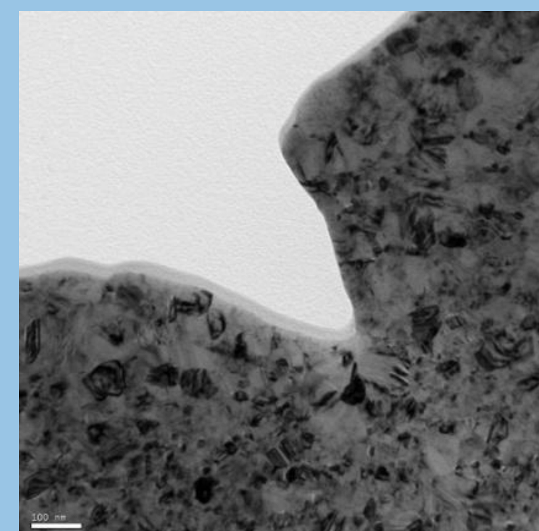
WHAT???
In-situ tests (tensional monotonic and cyclic) in the TEM with nc-Cu

WHY???
- To „see“ processes that dominate deformation in nc metals
- To test small sample volumes

FIB
Cutting free the MEMS and the sample



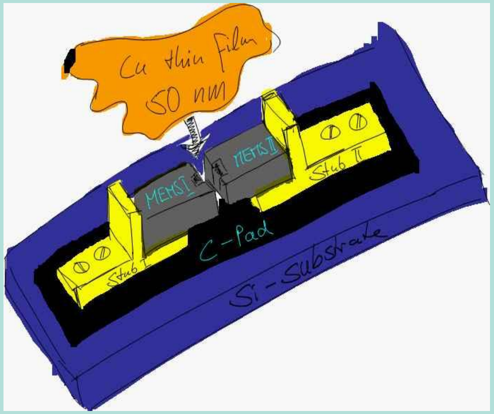
TEM
Tension testing
Fatigue testing



Samples – preparation and testing

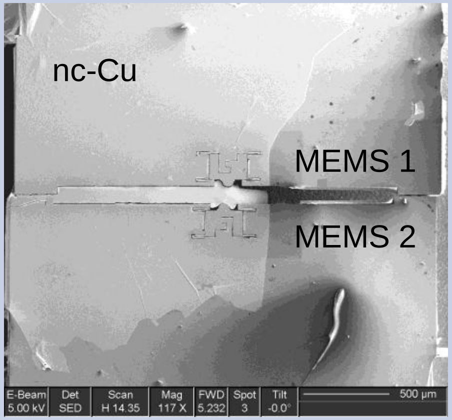
The 60 nm thick samples were electrodeposited on a mica plate with a 5 nm thick C-film between mica and nc-Cu.

1. Transfering the Film



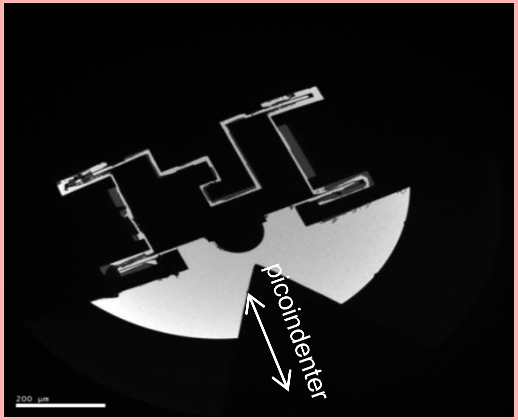
Film is going off the mica plate.
Floating on a liquid and sinking on the MEMS‘ testing section.

2. FIB - cutting



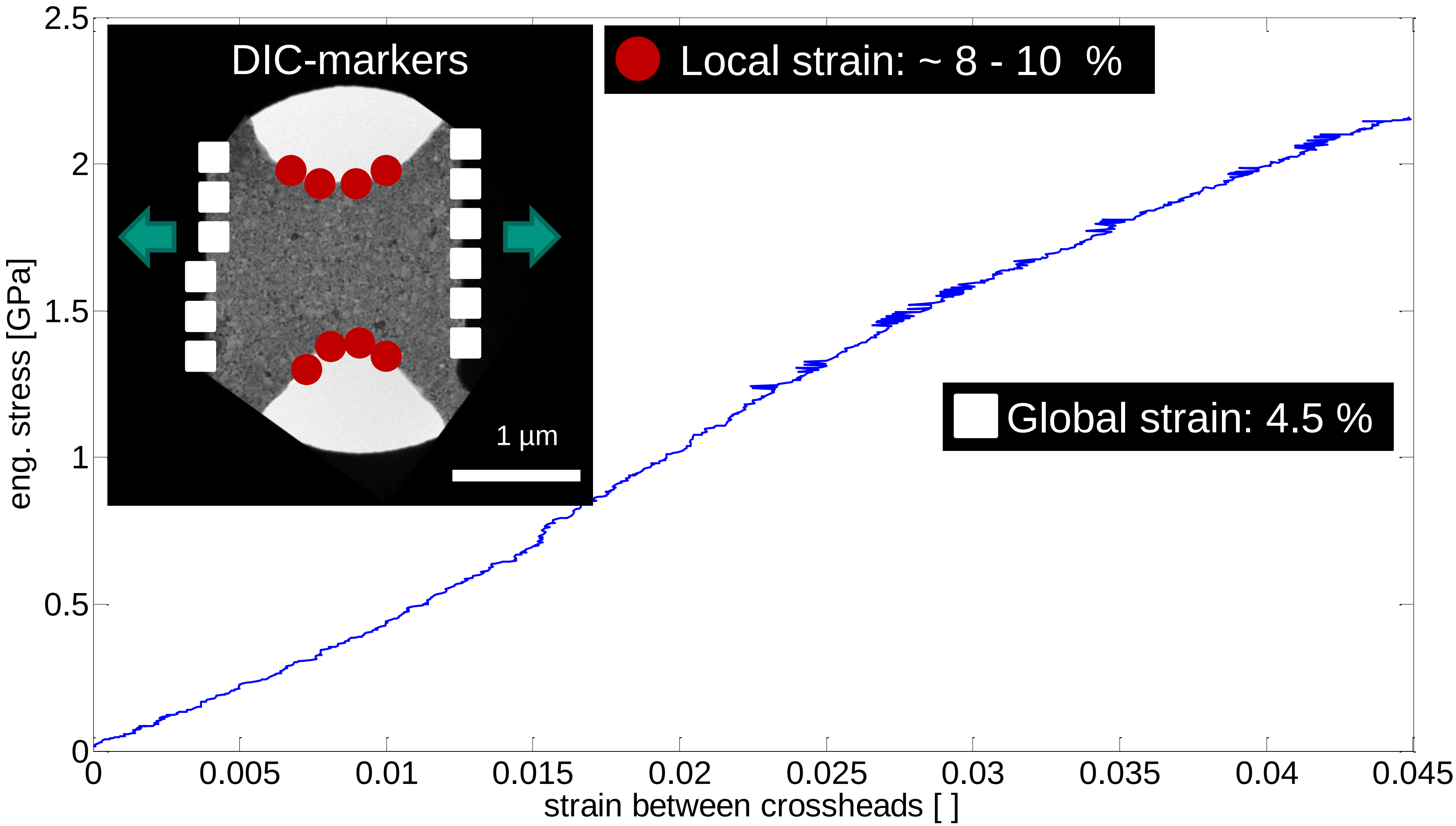
Film is drying and attached to the MEMS.
Cutting free the mechanics along edges.

3. TEM - testing



Picoindenter is pushing on the MEMS in the TEM.
Compression is trans-formed into tensional load and sample is strained.

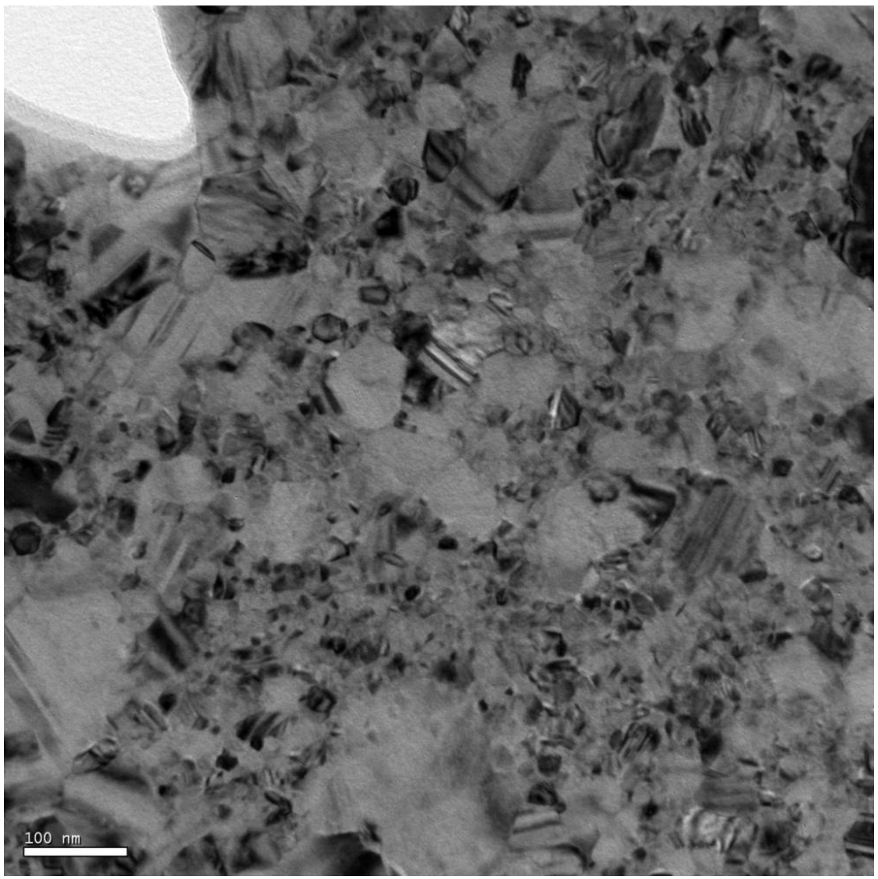
Stress-Strain Curve



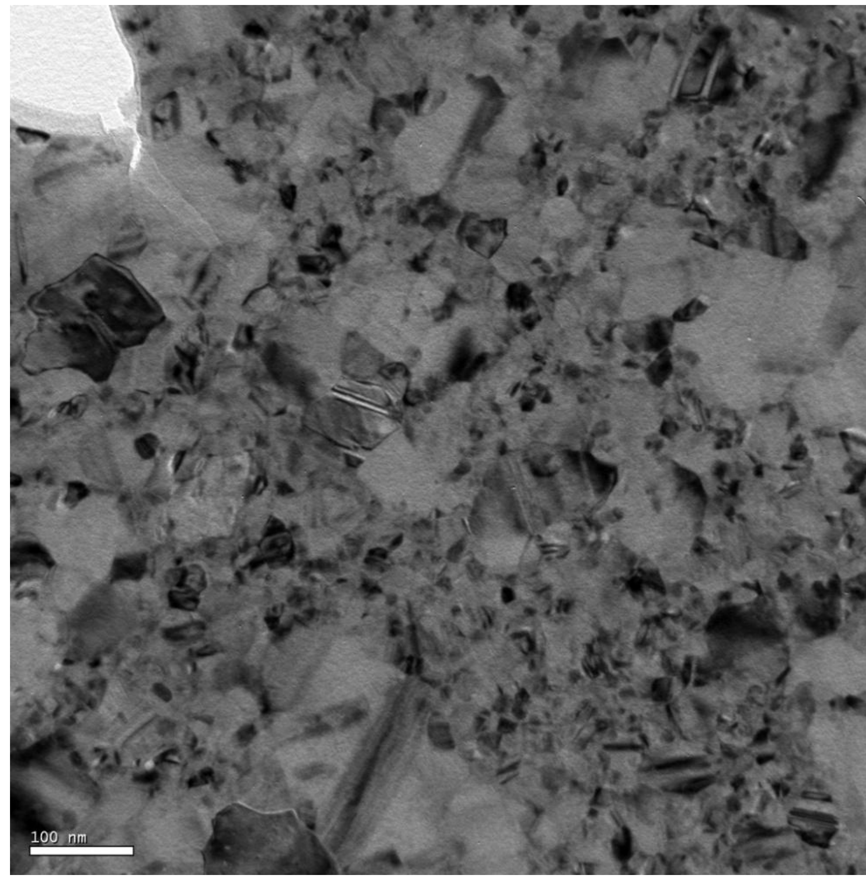
Thickness of nc-Cu ~ 65 nm →Young's modulus of ~ 50 GPa,

Fatigue of a notched sample

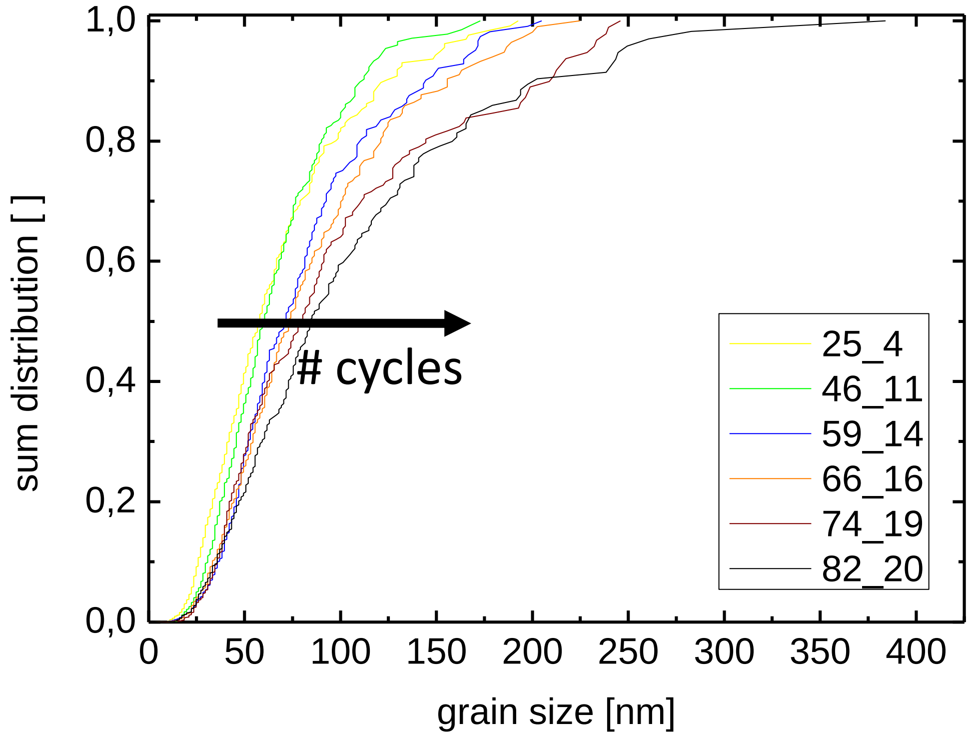
After 8th cycle



After 9th cycle
9th cycle



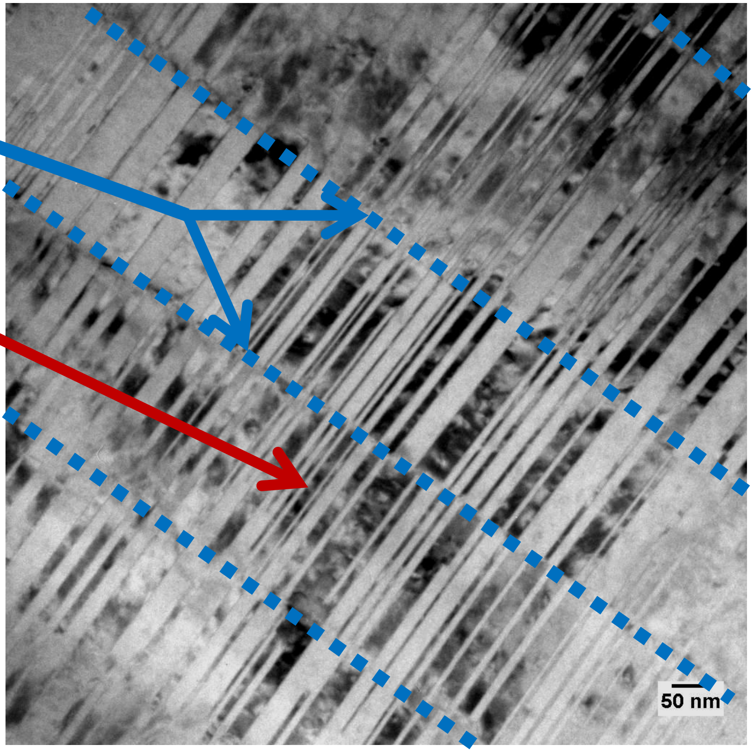
Grain coarsening during fatigue



Nanotwinned Copper

Ultra fine grained microstructure

Lamella distance of 5 – 50 nm



+ similar Hall-Petch-hardening than in nc

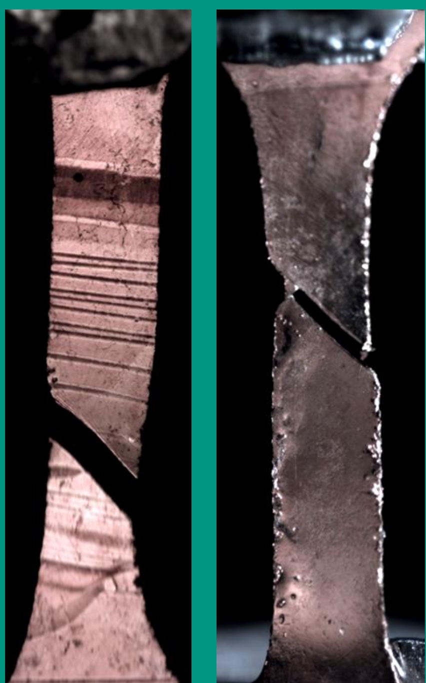
+ dislocation activity → ductility

- Deposition is time consuming (sputtered)

Mechanical testing: No necking / necking

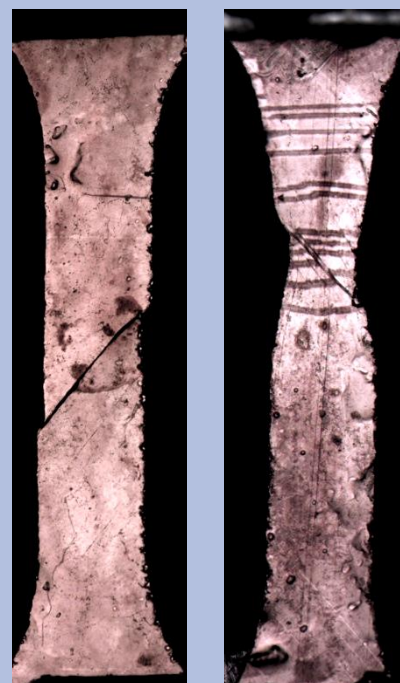
Tension

No necking / necking



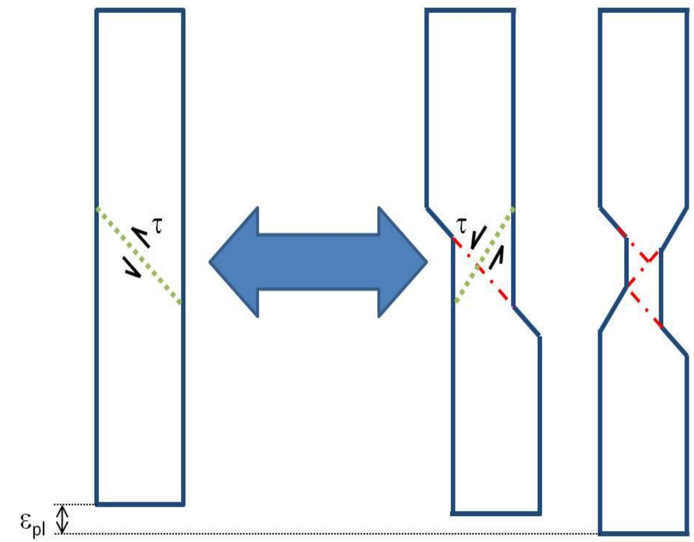
Fatigue

No necking / necking



Single slip
No necking

Multiple slip
necking

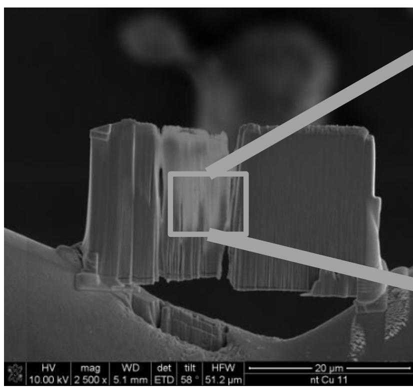


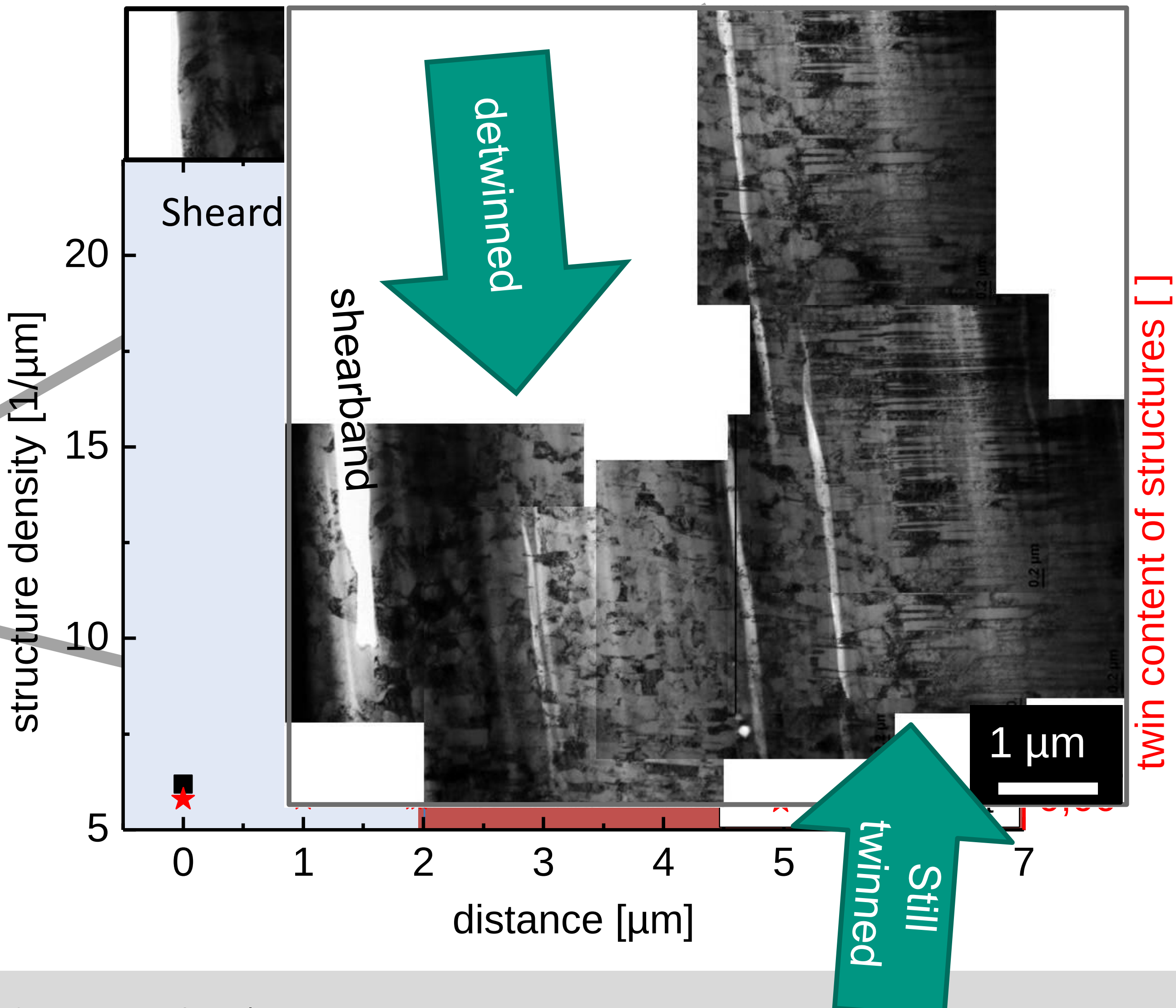
depending on diffusion / strain rate
→ Single / multiple slip

[Tests done by T. Kennerknecht]

Fatigued nt Cu

TEM lamella of the region around the shear band





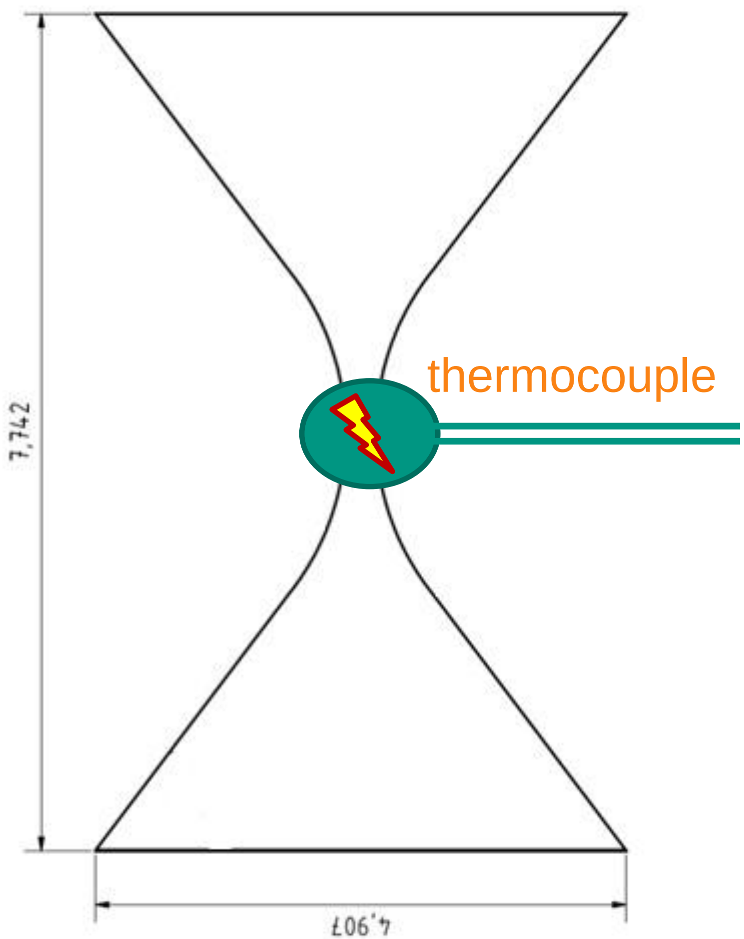
How much nonlinearity is in the deformation?!?

When will the sample break?!?

Fatigue test

Fourier Transform (FT) analysis

Motivation



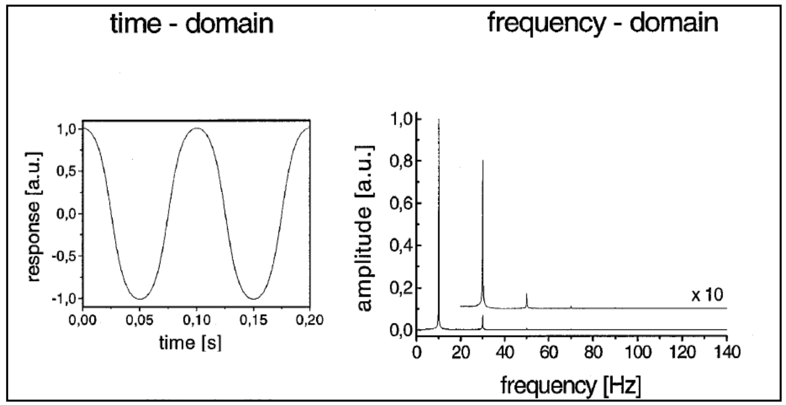
+ Quantitative evaluation of nonlinear fraction of fatigue signals (fingerprint)

+ Applicable for any kind of response signal e.g. torsion, bending, tensile...

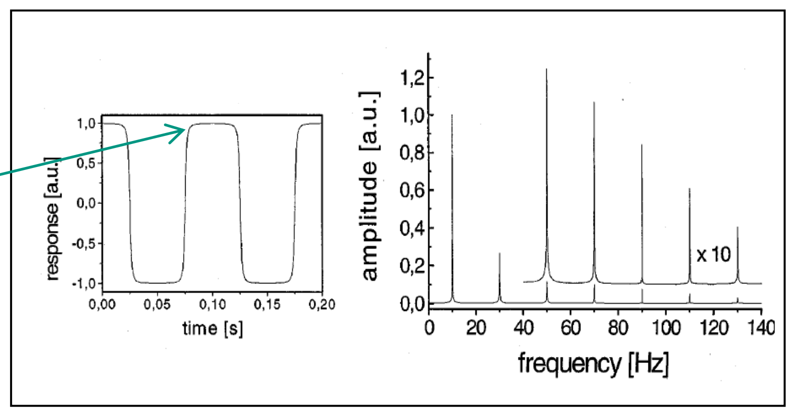
+ Contactless analysis of fatigue signals (valuable especially for nc and nt materials)

FT-Analysis of fatigue-signals

Fouriertransform-rheology with polymers –
harmonics are proportional to nonlinear fraction (Wilhelm)



Nonlinear fraction
~
Damage, degradation



[Wilhelm et al., ***Rheol Acta,*** 1998]

FT- Analysis: setup, parmeters, samples

GABO – Eplexor 150 N

Intent

Correlation: degradation ⟷ nonlinearity

Materials & samples

Nanocrystalline Ni (grain size < 100 nm)

Coarse grained Ni (conventional and MSG)

Cross section: ~ 0.056 mm^2

Acquisition parameter

Scan rate: 50 kHz

Box-oversampling: 250 pts.

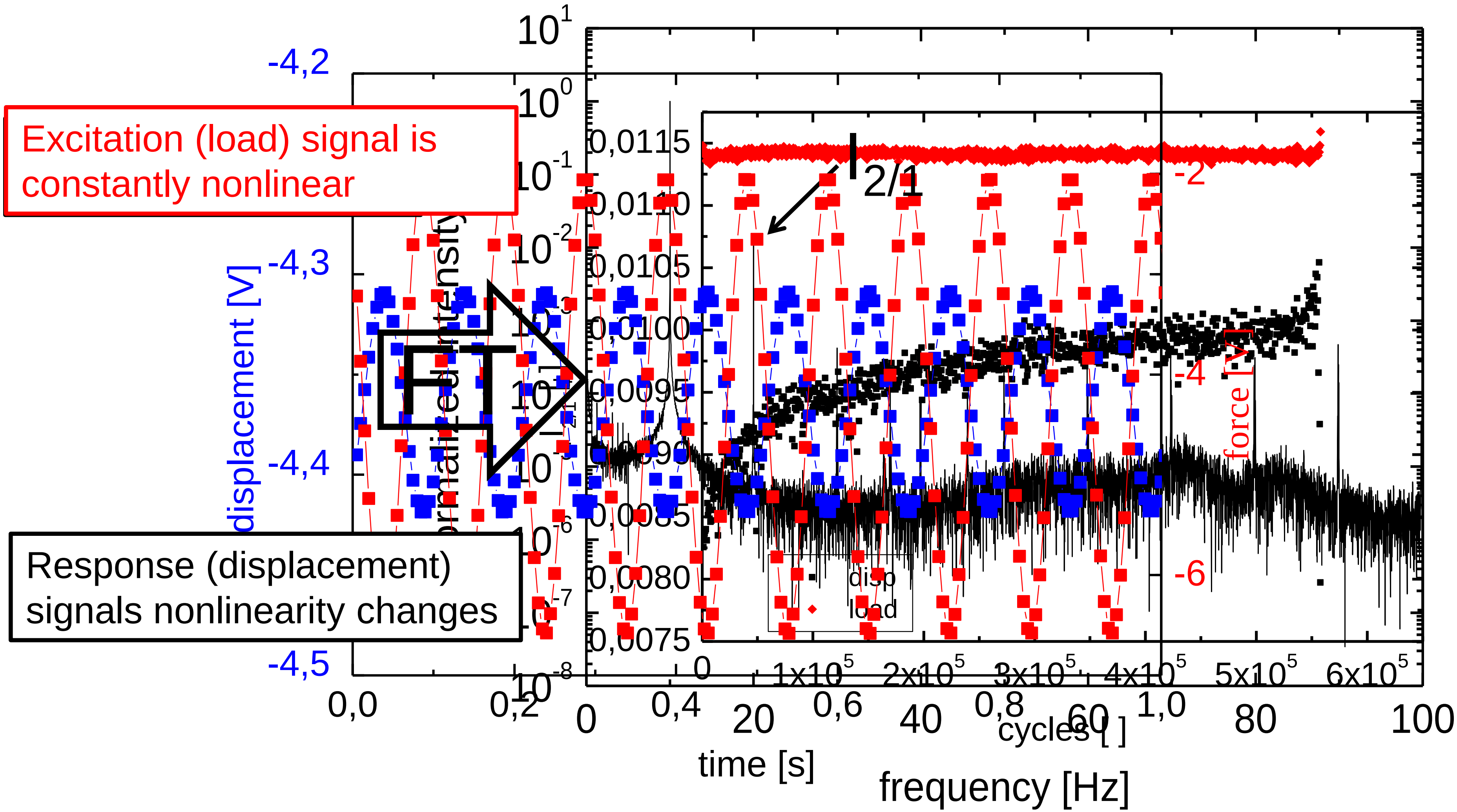
→ Effective sampling rate: 200 Hz

Acquisition time of segments: 60 s

Cycling parameter

- 10 Hz
- F_{stat} = 20 - 50 N and F_{dyn} = up to 23 N
- T = RT (-150 – 500 °C possible)

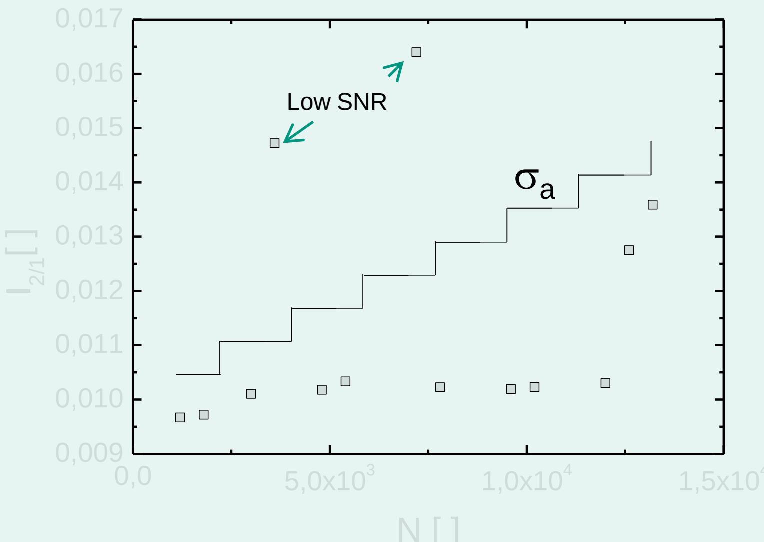
FT-Analysis of fatigue test with nc-Ni



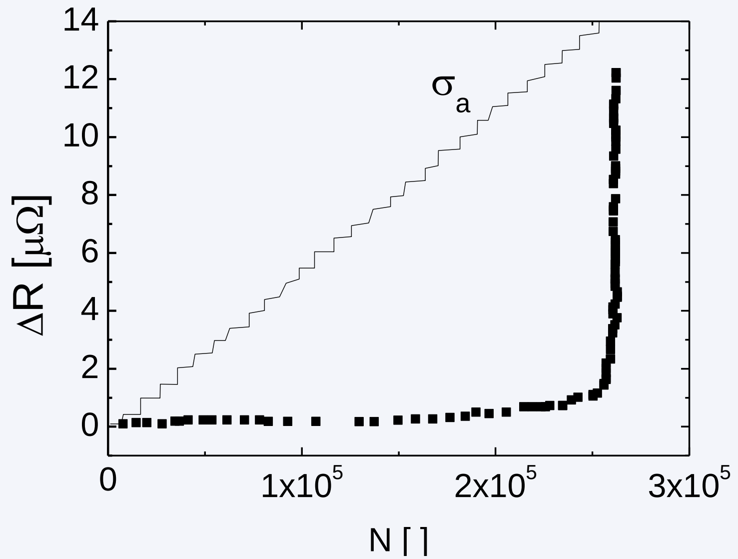
σ_m = 357 MPa σ_a = 179 MPa

FT-Analysis of load increase tests

Nanocrystalline Ni	Tempered steel
Load-increase-test	



$\sigma_m = 974$ MPa
$\sigma_a = 90 - 400$ MPa



[Starke et al., ***Mater Sci Eng***, 2009]

Summary

Karlsruhe Institute of Technology

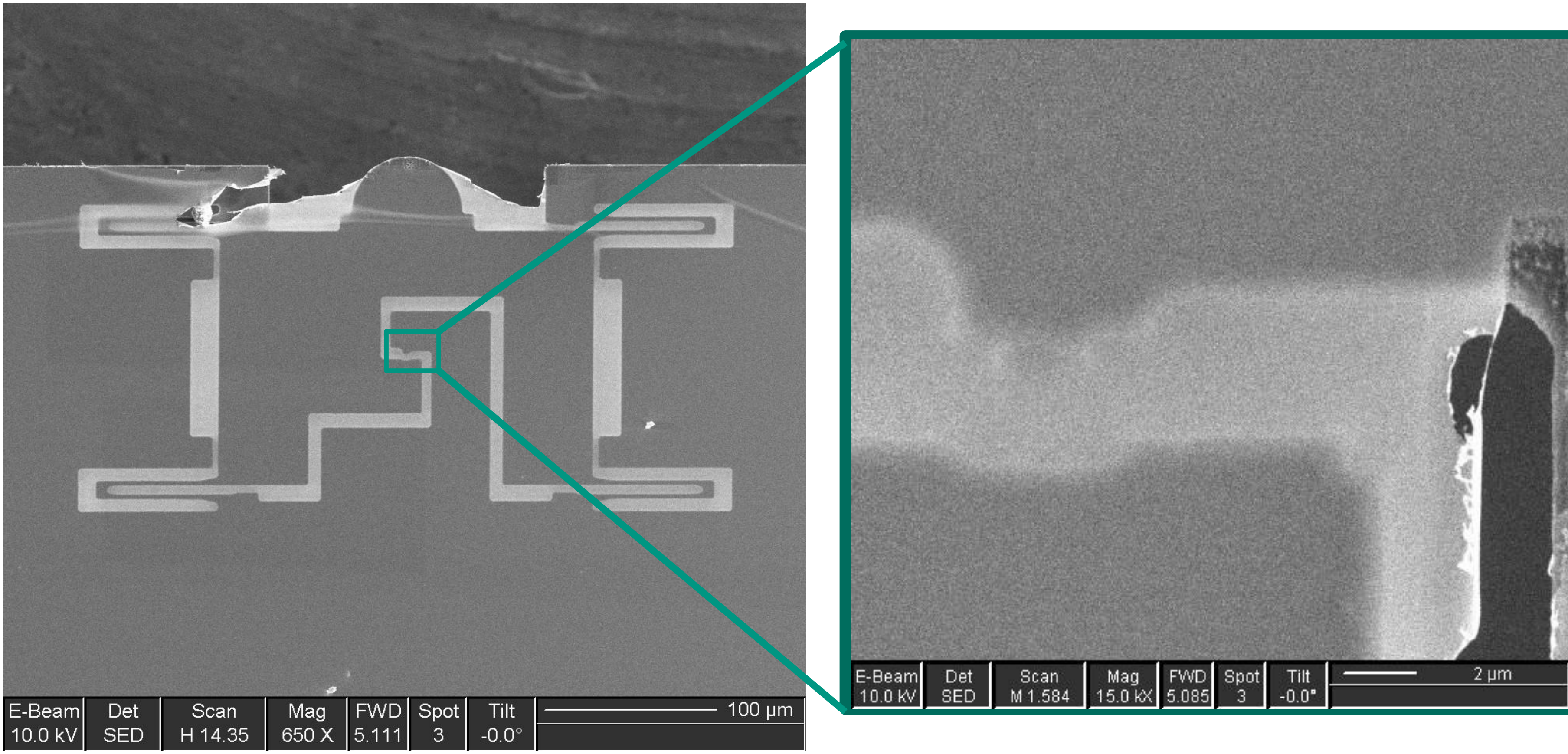
- Fatigue of nc-Ni: higher stress amplitudes between 10^4 and 10^7 cycles compared to cg-Ni
- TEM *in-situ* tensile and fatigue testing of nc-Cu: local strain of ~ 9 % and ultimate strength of ~ 2 GPa - grain coarsening in fatigue
- nt-Cu: multiple slip → necking. Detwinning in 2 µm around the slip band
- Analysis of fatigue signals with FT: increase of nonlinearity at the end of fatigue life could be used for lifetime prediction (fatigue criteria)

Acknowledgement

- Claire Cishholm, John Turner, Hua Guo for their help at the NCEM
- Tobias Kennerknecht, Ewald Ernst for their help with the bending setup

Thank you for your attention!

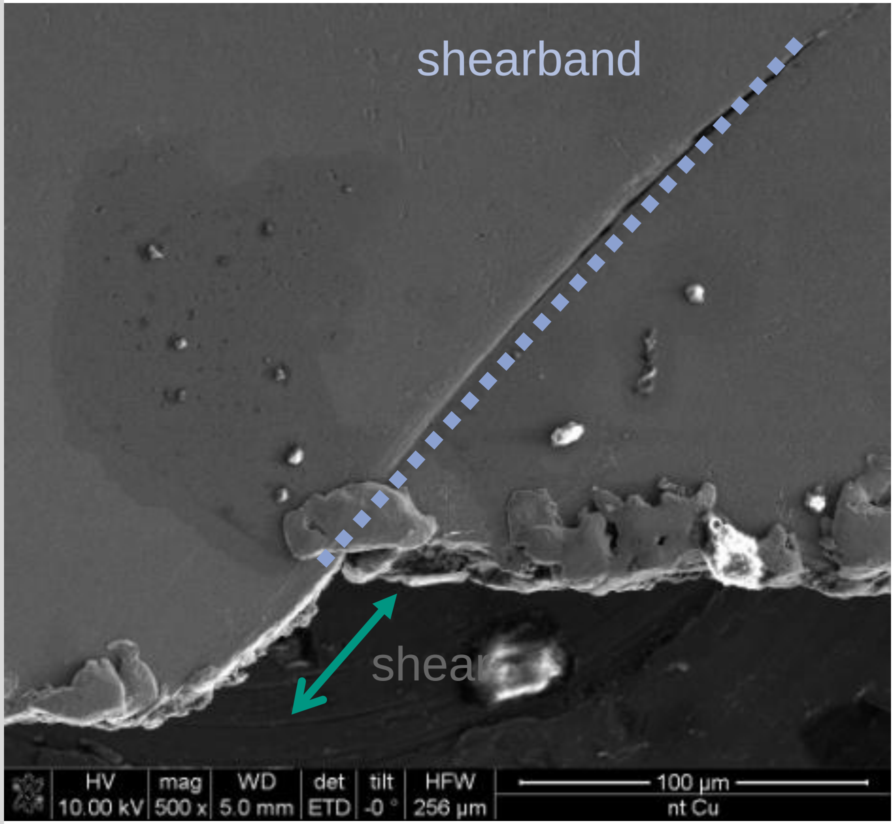
Cutting the Film



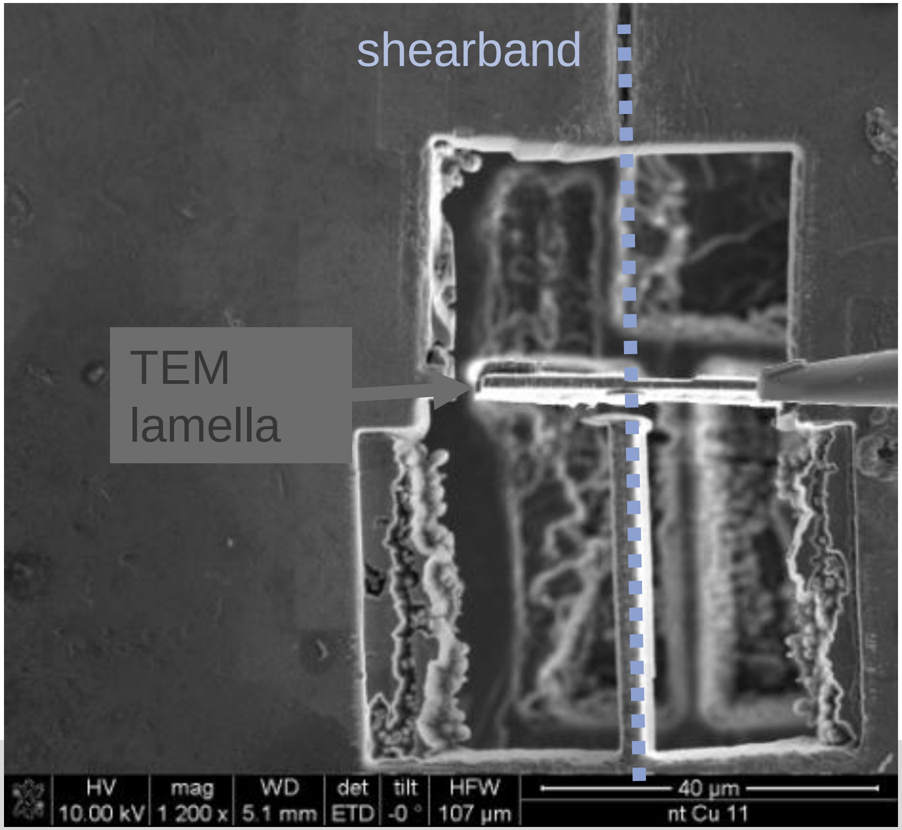
Off sample: 10 kx (reduced window), 1 - 3 nA

Close to sample: 15 kx (round pattern), 50 - 100 pA

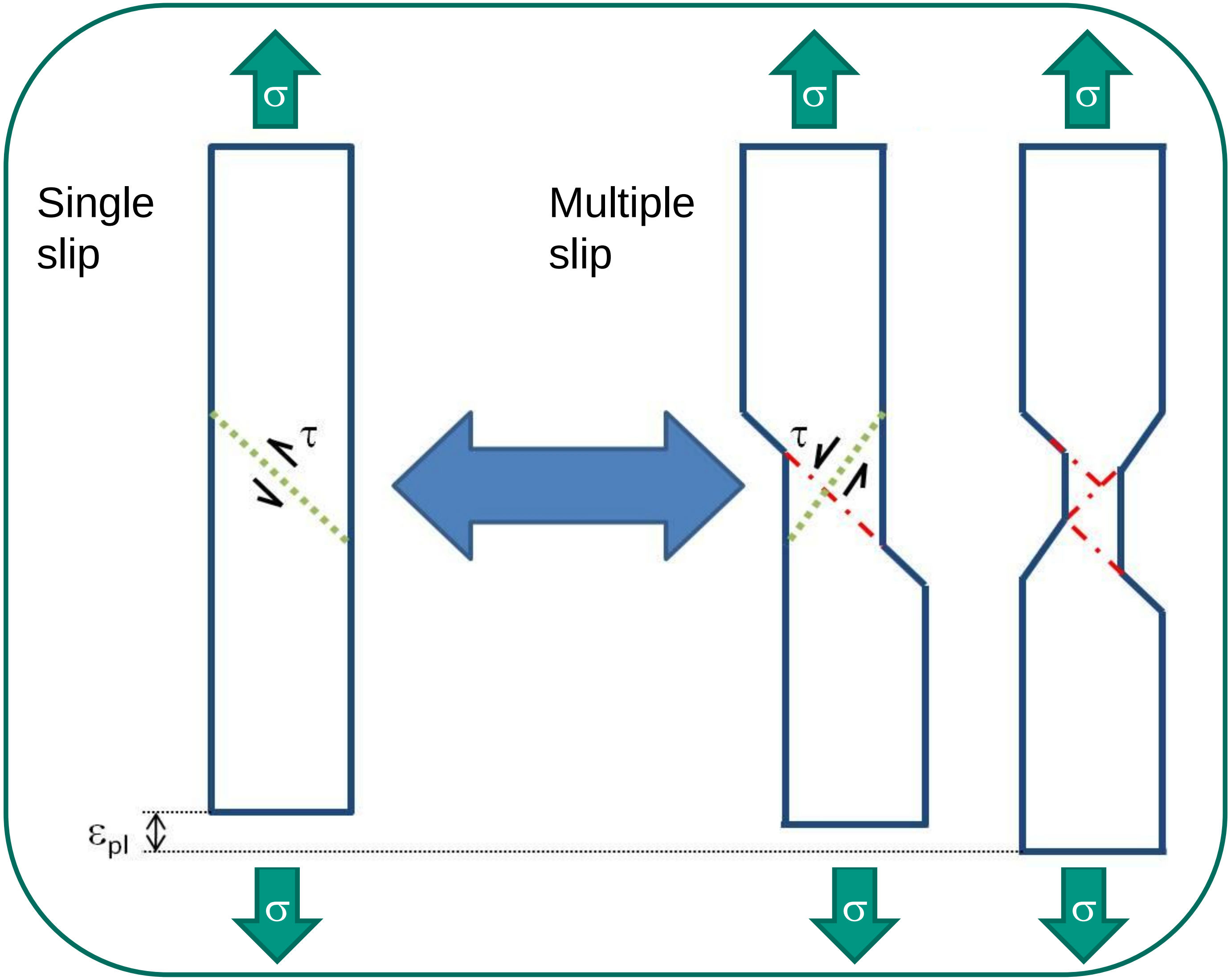
nt Cu 11



TEM Lamella with the shearband in the middle



Multiple shear - necking

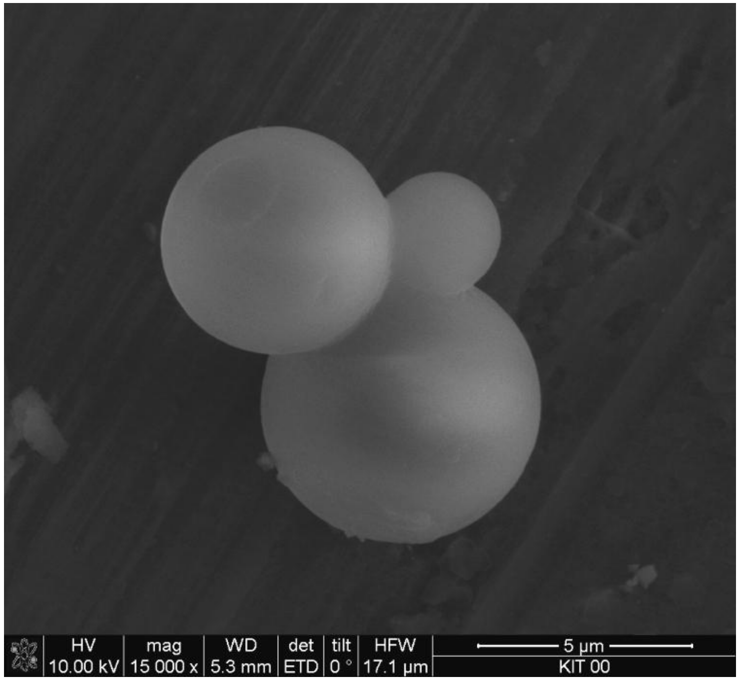


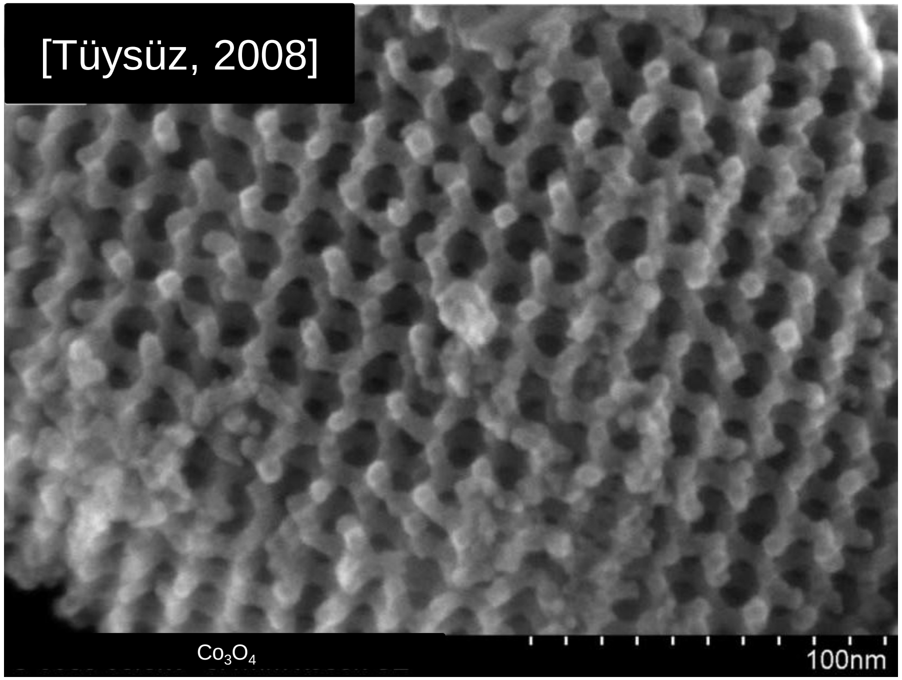
Dislocation source exhaustion→ multiple slip

necking

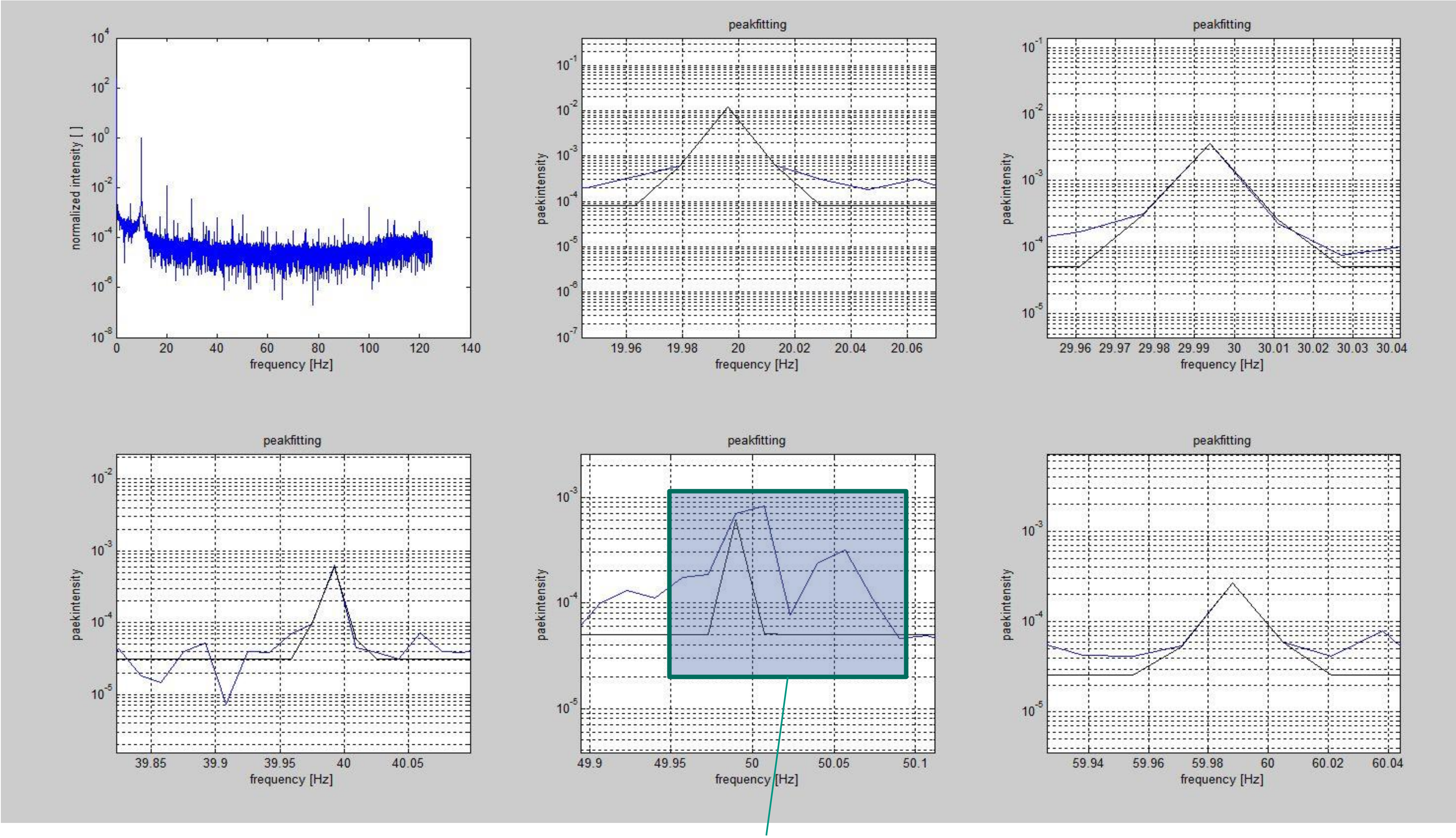
possible project – nanoporous materials

Nanoporous Co_3O_4 and ordered silicate hard template materials have promising properties for applications in kathalysis industry, to investigate their mechanics:
- in-situ compression tests in the SEM
- ex-situ compression tests in the nanoindenter





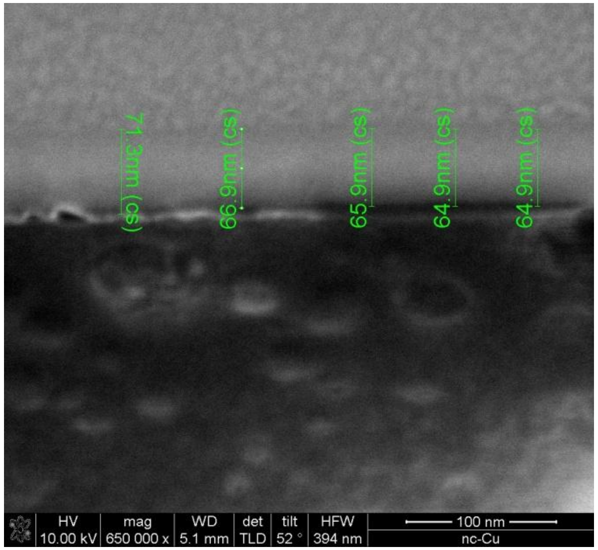
FT and peaks of 5 harmonics



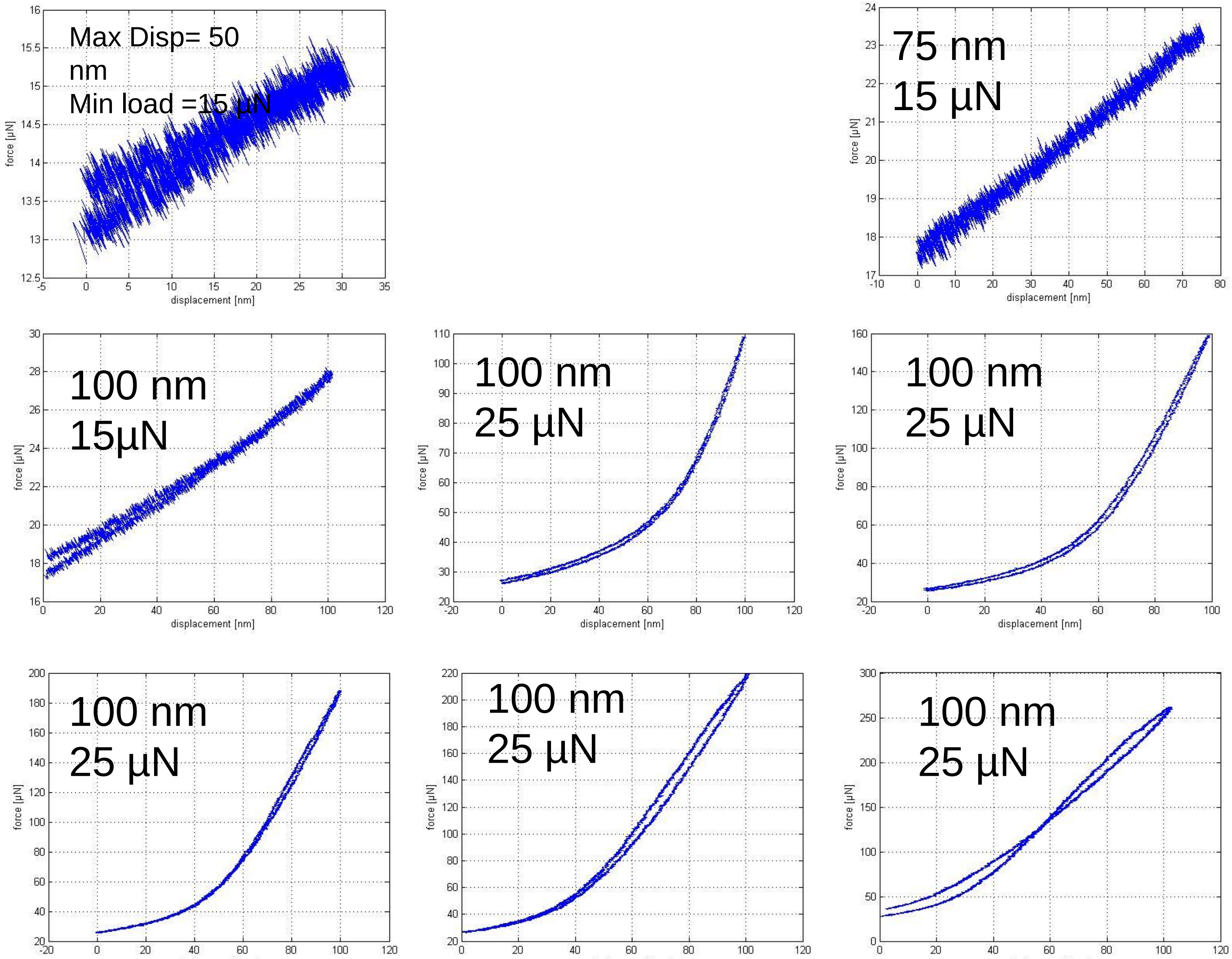
Frequency oszillations of 0.1 %, 0.05 Hz are normal (RWE)

Nonlinearity parameter

$$\beta_{\text{expt}} = \frac{8A_2}{A_1^2 k^2 x}$$

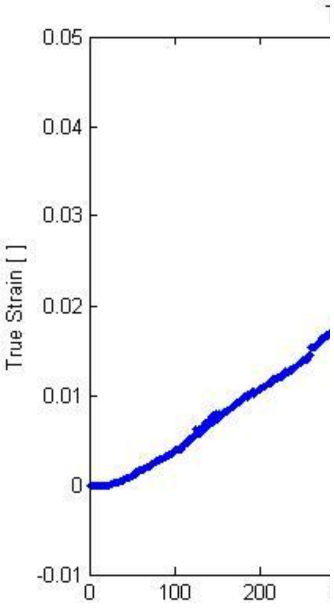


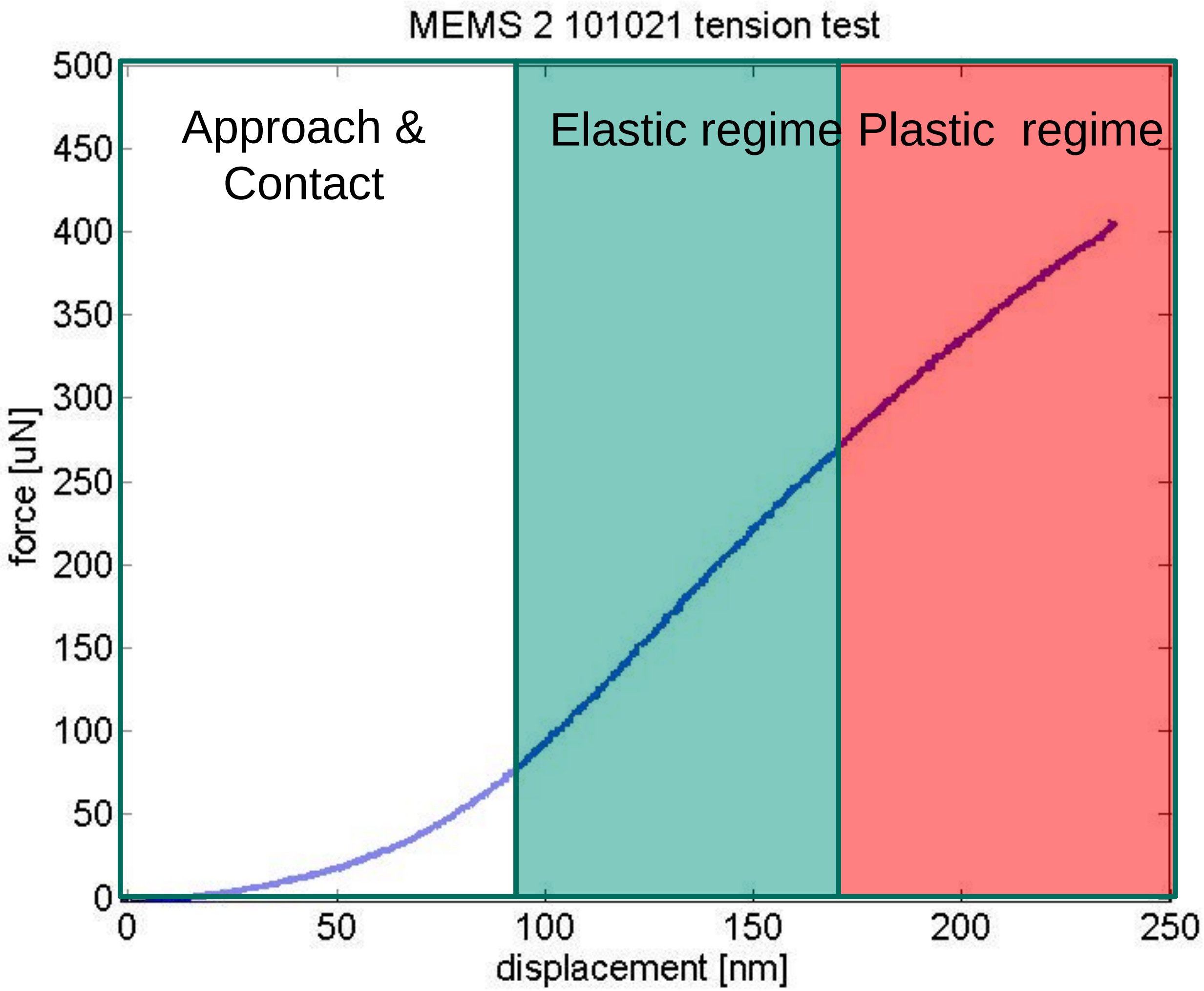
Loading curves



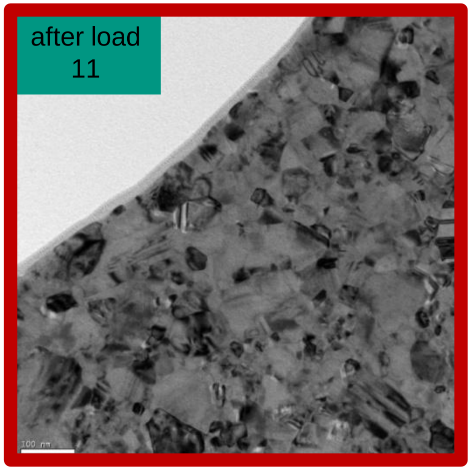
results

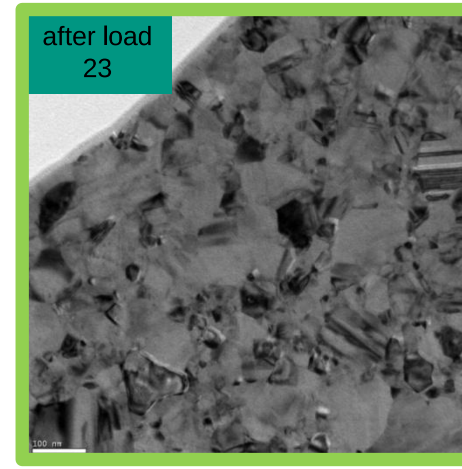
Strain

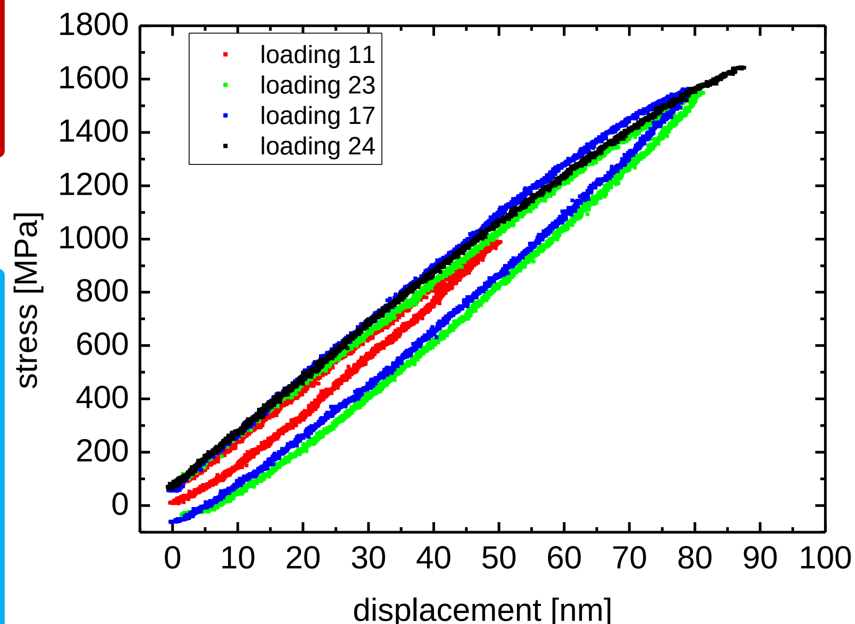


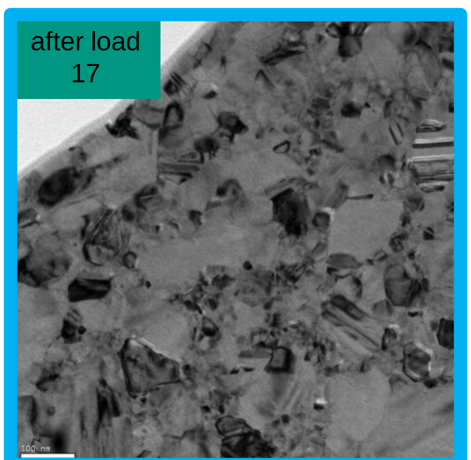


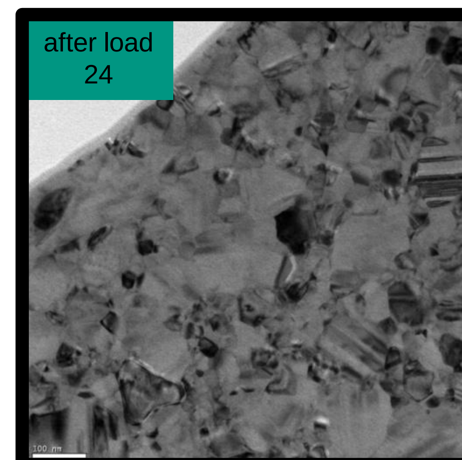
Grain coarsening Pos 1



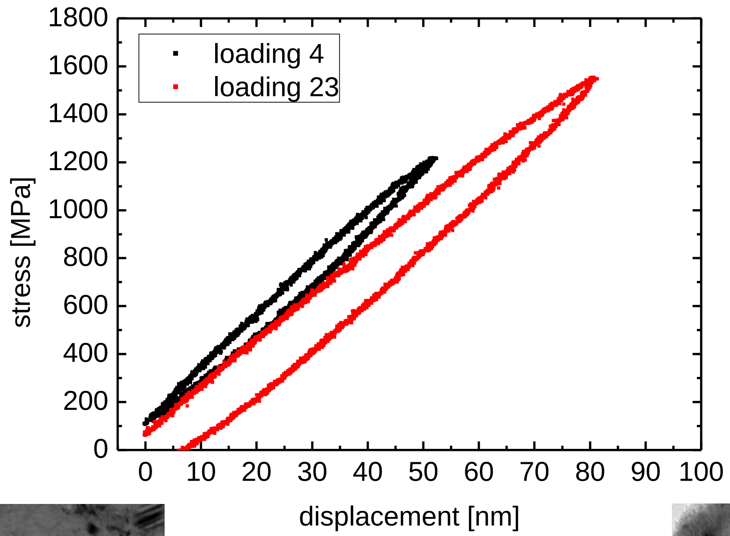


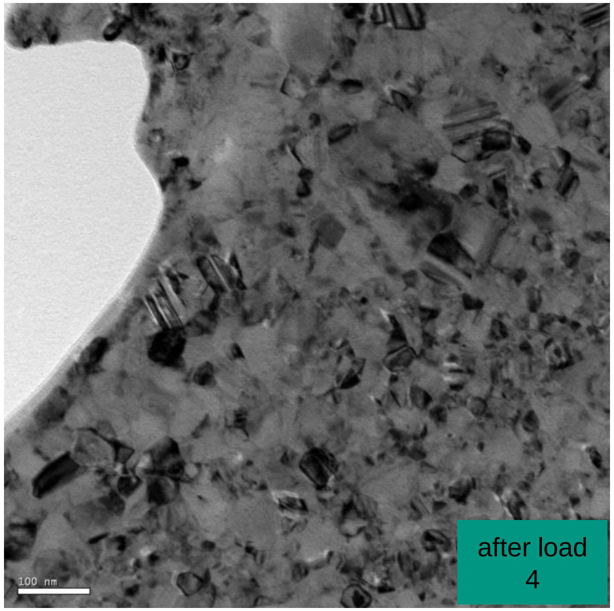


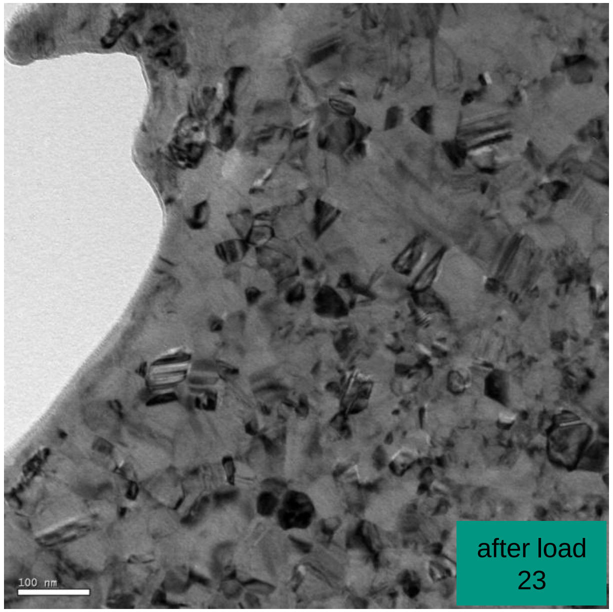




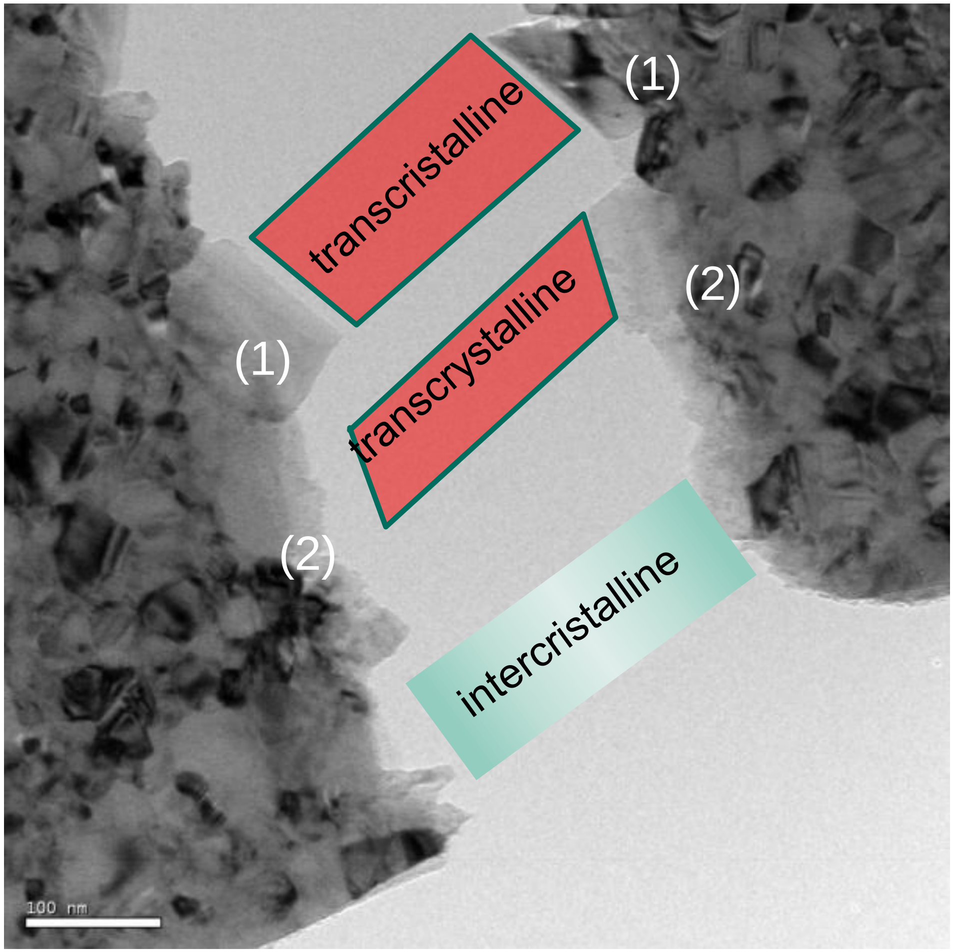
Grain coarsening



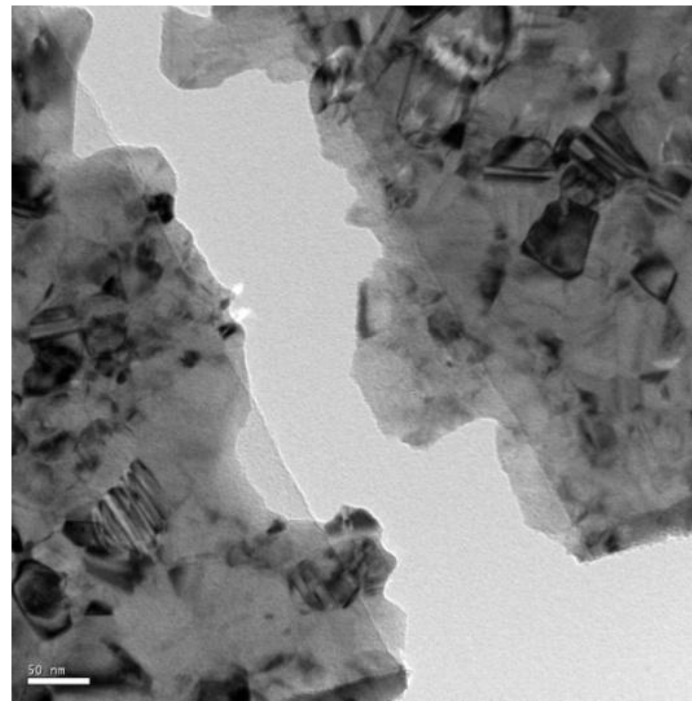




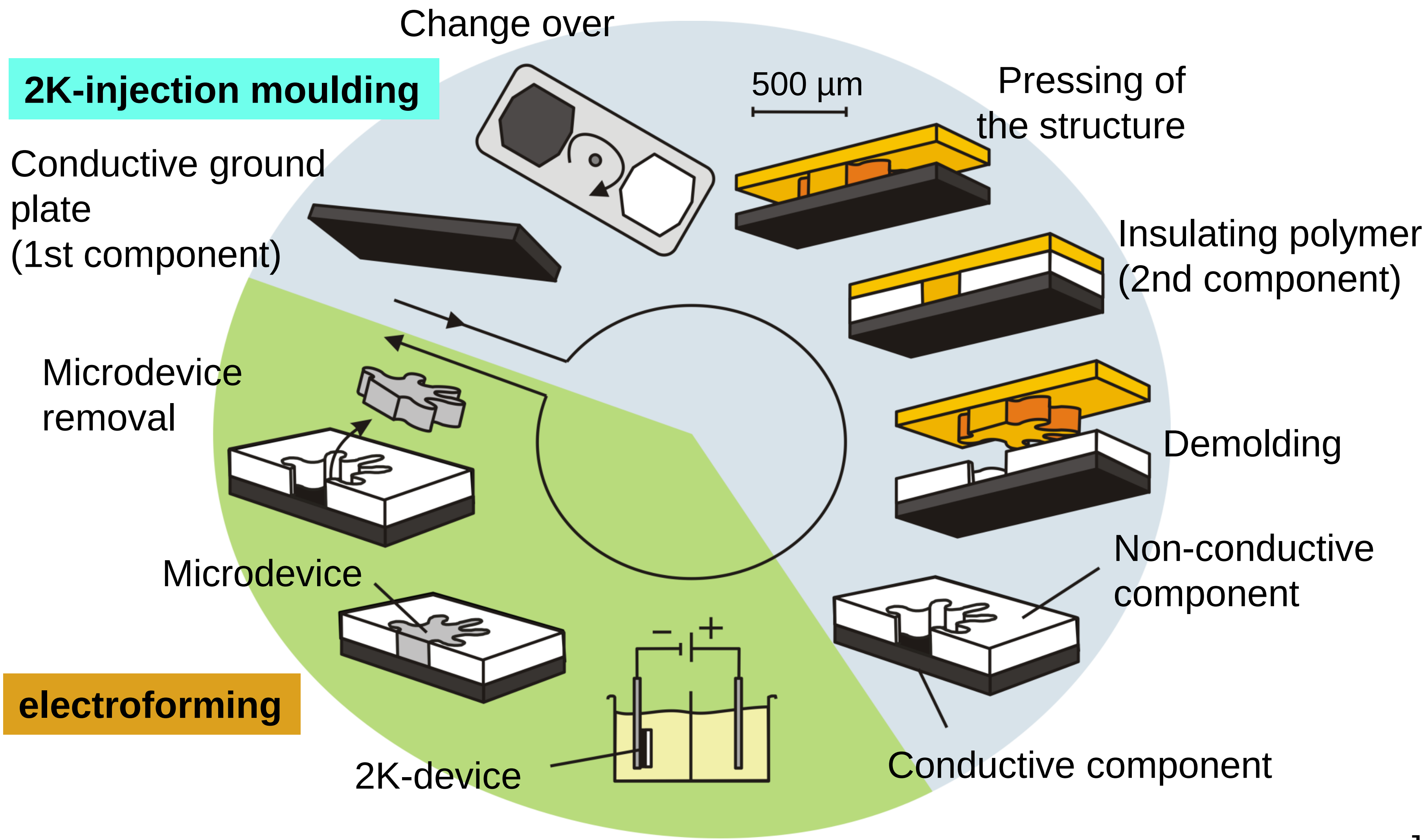
Fracture Morphology nc-Cu



Transcrystalline cracking in big grains (ufg); d > 100 nm
Intercrystalline cracking in smaller grains (nc); d < 100 nm



Mehrkomponenten Spritzgießen und Galvanoformung (MSG)

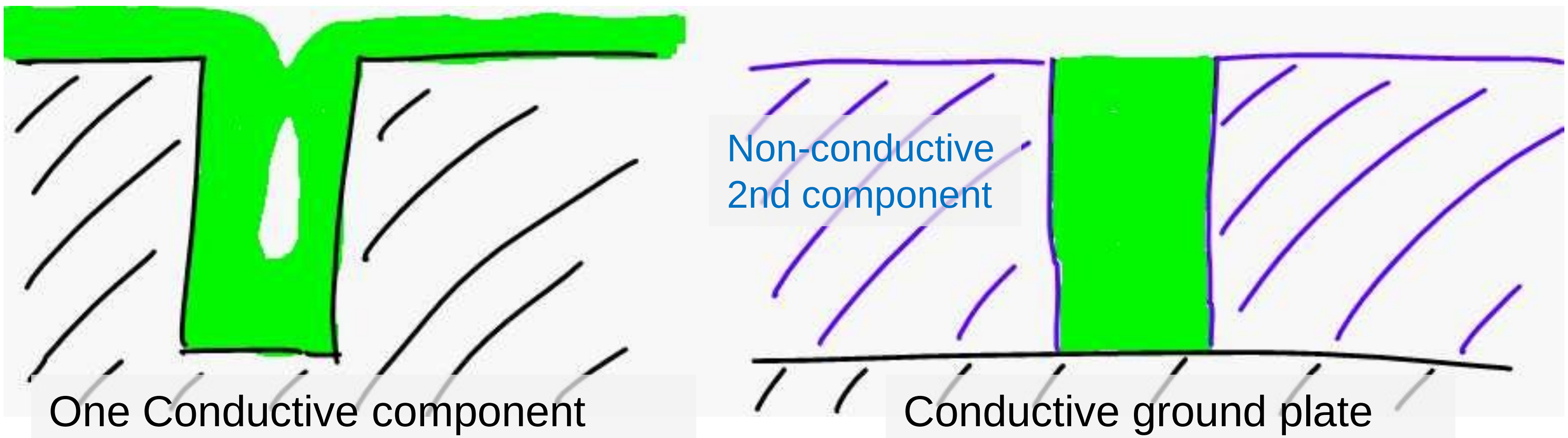


[Jürgen Prokop]

MSG - Motivation

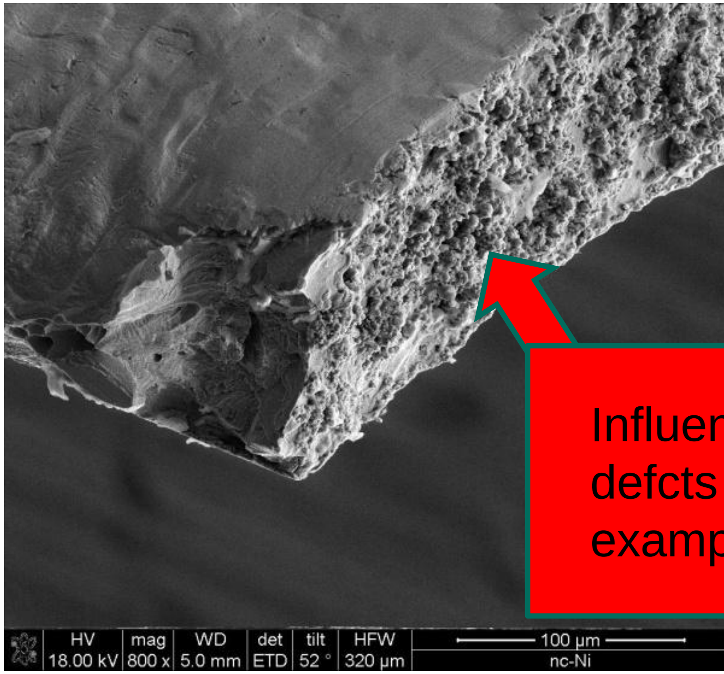
Conventional electrodepositing

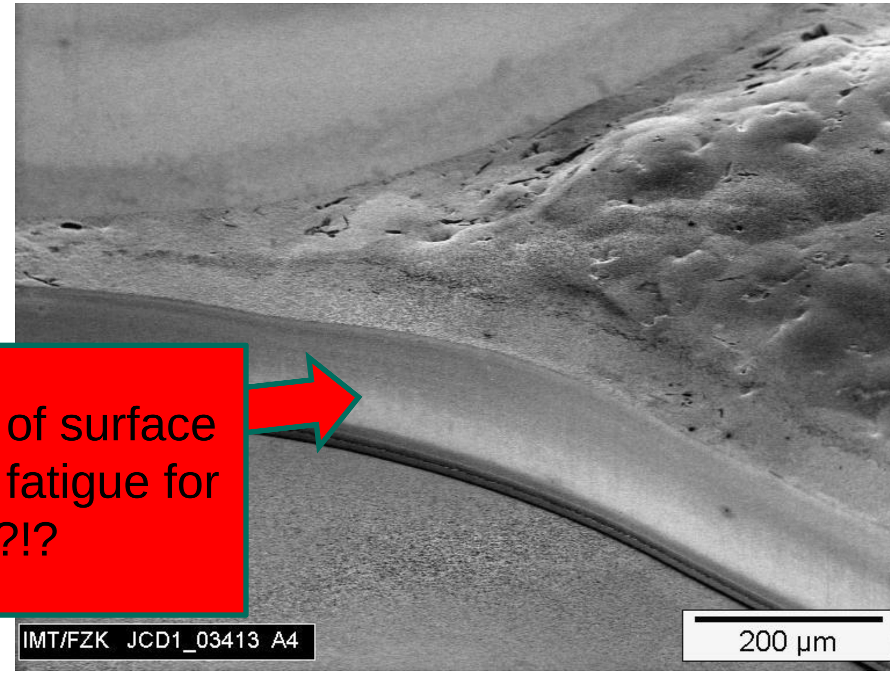
MSG - method



Benefit: no overgrowing and no pores in microparts with small cavities

Comparison of surface quality





Influence of surface defcts on fatigue for example ?!?

EDM-sample

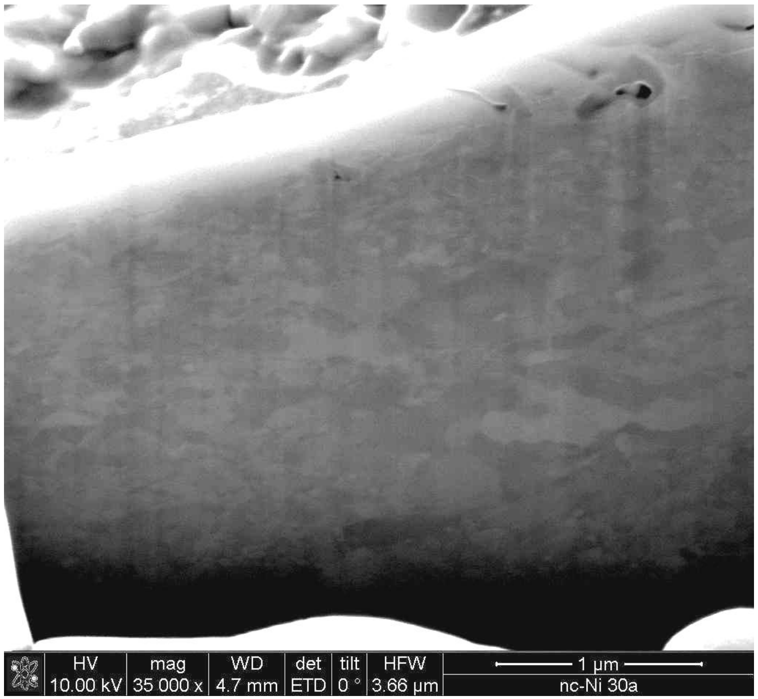
Electrodeposited
Ni-5 at% W (LIGA) [Jürgen Prokop]

Benefit: Less defects in surface
No surface layer (oxides, hydroxides)
No heat affected zone

Grain coarsening - coalescence

Grain coarsening could be necessary to enable dislocation structures, which leads to failure

Grain rotation could lead to coalescence (grain coarsening).



[Meyers ***Progr Mater Sci*** 2006]