

Ringvorlesung Sensorische Materialien

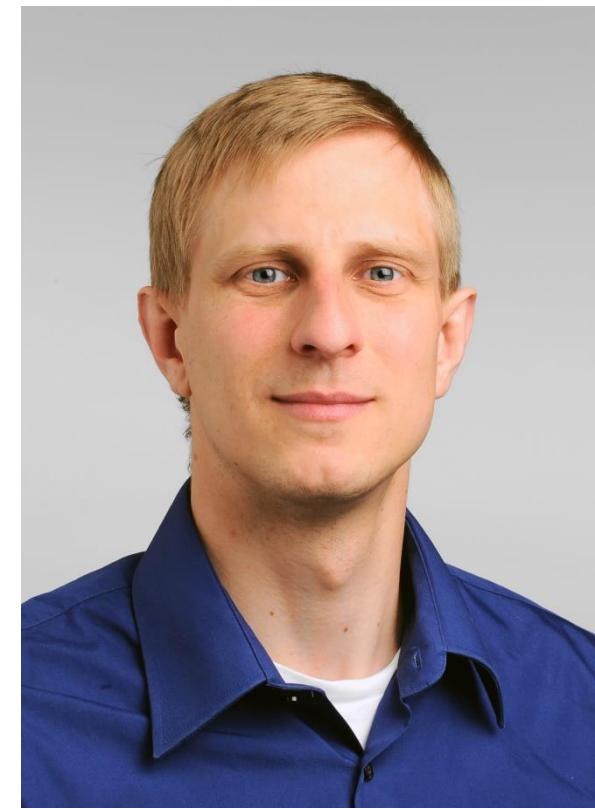
Funktionskonforme Sensorintegration mit magnetostriktiven Partikeln

**Steffen Opitz, Peter Wierach, Marcus Kubicka, Thorsten Mahrholz,
Alexandra Kühn, Michael Sinapius**

Zu meiner Person

Steffen Opitz:

- Studium der Mechatronik an der Otto-von-Guericke Universität Magdeburg
- Promotion mit dem Thema: Funktionskonforme Sensorintegration in hoch belasteten, adaptiven, umströmten Strukturen
- Mitarbeiter am Institut für Faserverbundleichtbau und Adaptronik des Deutschen Zentrums für Luft- und Raumfahrt
- Stellvertretender Abteilungsleiter der Abteilung Multifunktionswerkstoffe
- Mitarbeit in der Forschungsgruppe 3022: Ultraschallüberwachung von Faser-Metall-Laminaten mit integrierten Sensoren



Institute of Composite Structures and Adaptive Systems

Director: Prof. Dr.-Ing. Martin Wiedemann
Vice Director: Prof. Dr.-Ing. Peter Wierach



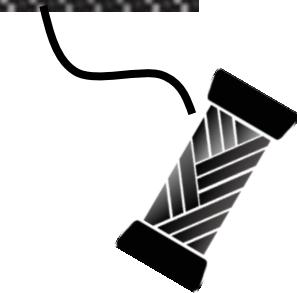
Knowledge for Tomorrow



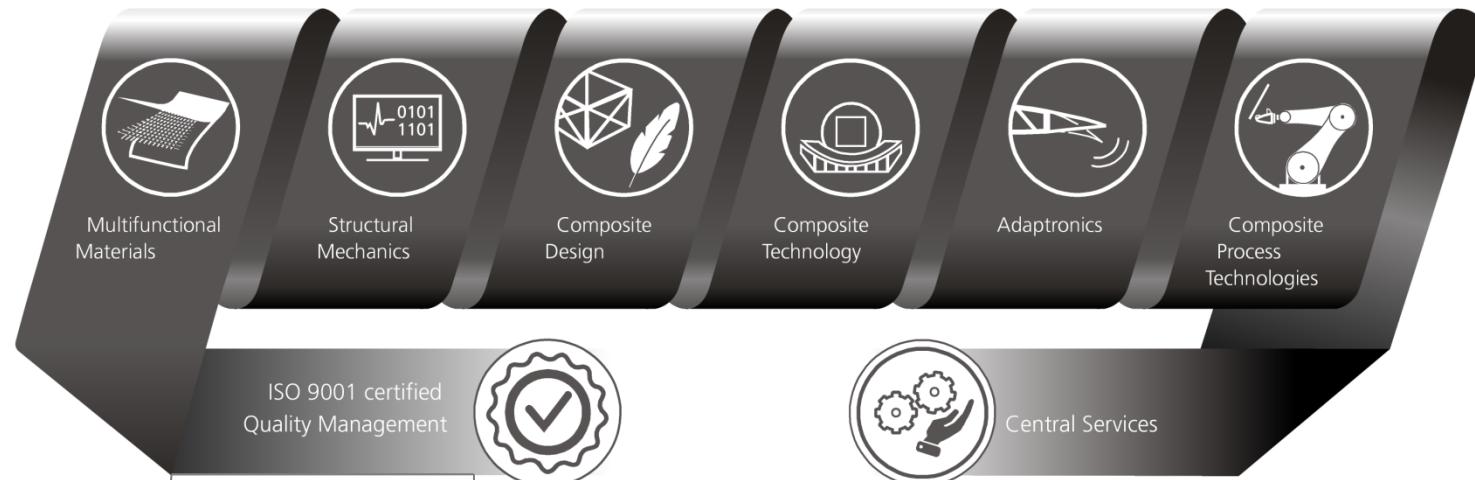
Our Mission

Design and realize adaptable, efficiently manufactured and tolerant lightweight systems. We do research on:

- proper lightweight design
- cost efficiency
- improved functionality
- higher degree of comfort
- environmental protection



The institute in detail – six scientific departments, certified laboratories





More functionalities in the materials

Our research focusses at:

- Composite materials & Nano composites
- Smart Materials
- Structural Health Monitoring (SHM)
- Characterization of materials

We are concentrating on the development, characterization and qualification of composite materials with superior properties and new functionalities



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Knowledge for Tomorrow

Outline

- Introduction and motivation
- Idea and potential
- Challenges and approach
- Material processing and manufacturing of specimen
- Optical assessment of specimen
- Test setup and experimental results
- Conclusions and future work



Introduction

The ultimate Goal

Ultimate smart material properties:

- Sensor capabilities
- Actuator abilities
- Other functions
(conductivity, good FST properties,...)
- High strength / robustness
- High stiffness
- Lightweight



Introduction

The ultimate Goal

Ultimate smart material properties:

Carbon fiber reinforced polymers

- Sensor capabilities ?
- Actuator abilities
- Other functions
(conductivity, good FST properties,...)
- High strength / robustness ?
- High stiffness ✓
- Lightweight ✓

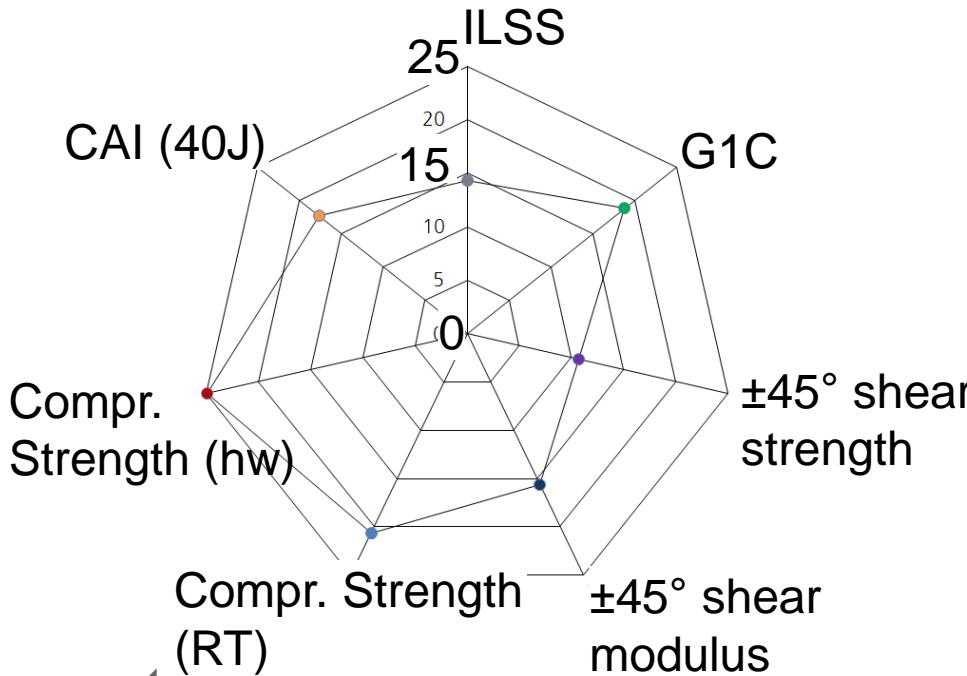


Introduction

Potentials of matrix modification

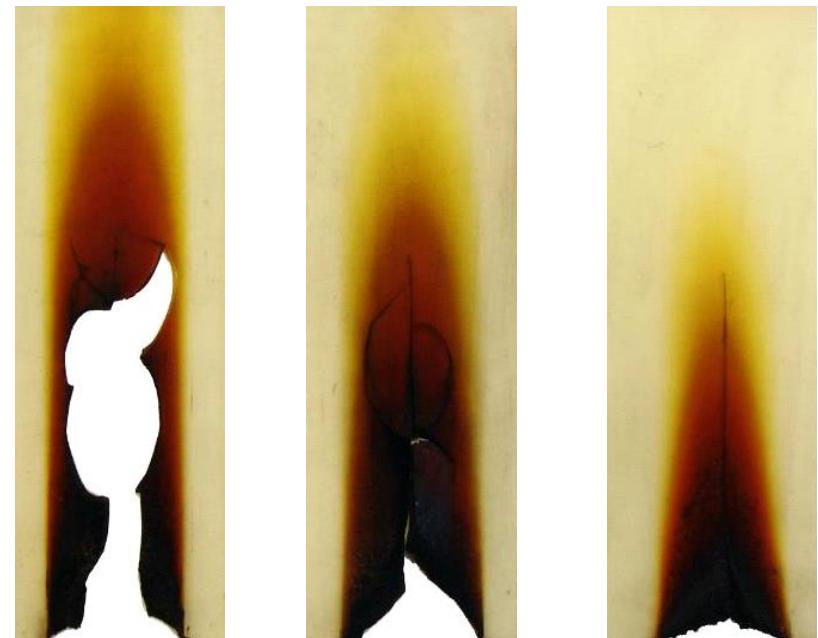
Mechanical properties

- Boehmite nano particles
- Nano-composite with 15 wt.-%



Integrated fire protection

- Epoxy resin modification with ATH (Aluminumtrihydroxid) nano particles



5% ATH
Nano particles

10% ATH
Nano particles

20% ATH
Nano particles

Introduction

The ultimate Goal

Ultimate smart material properties:

- Sensor capabilities
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(conductivity, good FST properties,...)
- High strength / robustness
- High stiffness
- Lightweight

Carbon fiber reinforced polymers

?

?

✓

✓

✓

✓

Composite materials offer:

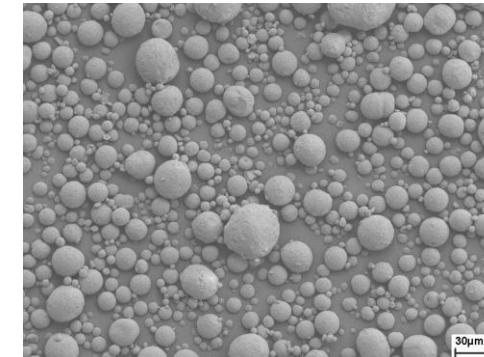
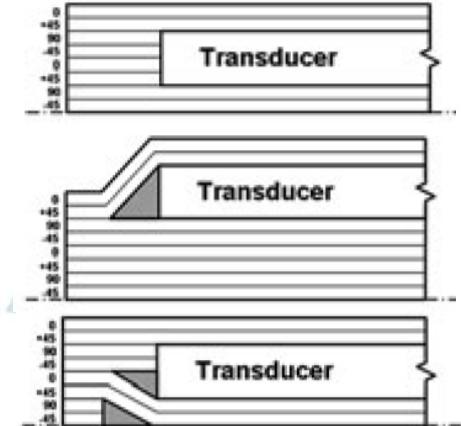
- Excellent strength to density ratio
- Possibility for the integration of additional functionalities by
 - Modification of the different material phases
 - Integration of new material phases in the material system



Introduction

Integration of smart / active materials

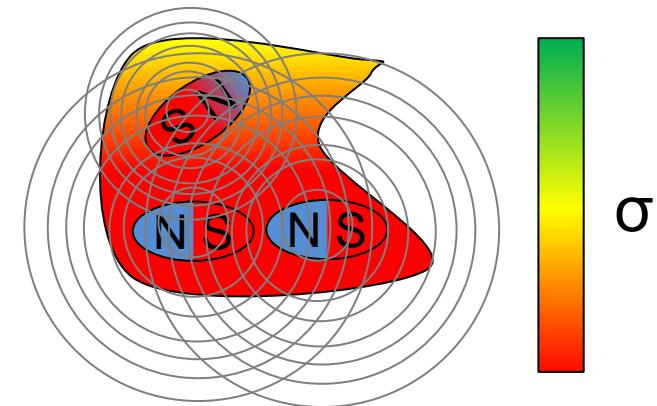
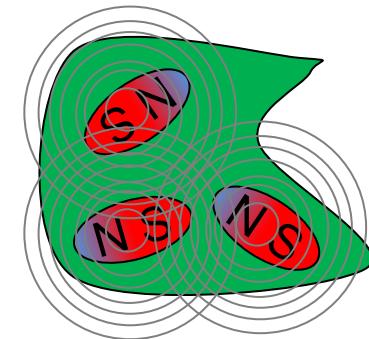
- Integration / Application of smart materials as foils/plates
 - Discontinuity in stiffness
 - Weakening of the structure?
- Integration of fibers
 - Complex manufacturing
 - Issues of contacting
 - Limited interfacial surface with the resin
- Integration of micro- or nano-scaled particles
 - Knowledge from previous works
 - Structural conformable
 - Functional conformable?
 - Filtering during injection?



Idea and potential

Basic idea

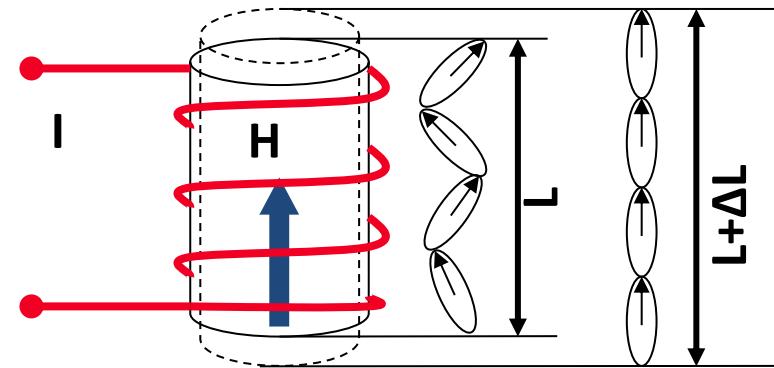
- Integration of sensor capability to detect stresses in the material
- Modification of the resin with:
 - Piezoelectric particles
 - Magnetostrictive particles→Distributed sensor



Idea and potential

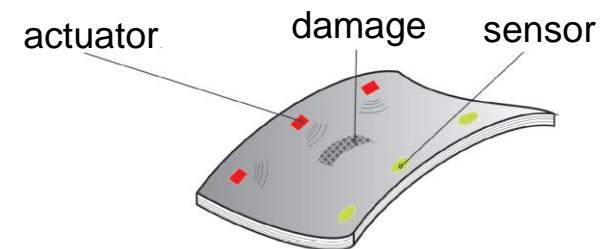
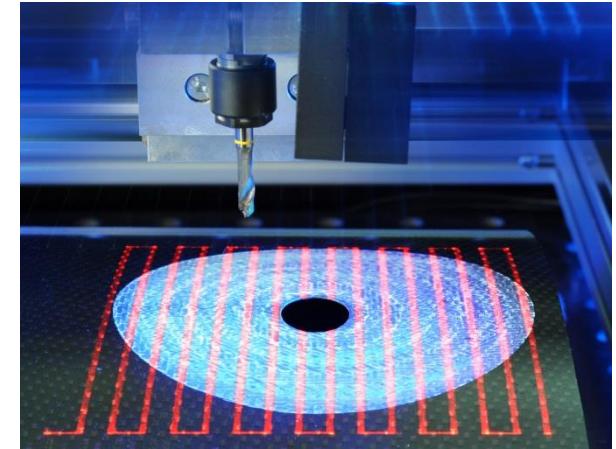
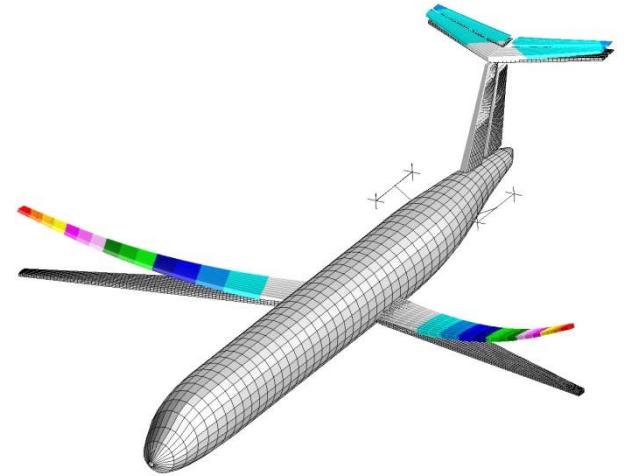
Basic idea

- Embedding of micro- or nano-scaled magnetostriuctive particles into the resin
 - Small size particles → large surface Area to introduce the loads into the particle
 - Focus on Terfenol-D
- Change of magnetic flux density due to strain
→ Villary effect (inverse magnetostriiction)



Idea and potential ... for transport and mobility

- Assessment of:
 - Structural loads
 - Loads during operation
 - Condition based maintenance
 - Residual stresses
 - Design optimization
 - Process optimization
 - Quality of bonds
 - Repair
 - Detection of kissing bonds
- Improvement of health monitoring systems
- Distributed over the whole structure
- Without any negative impact on the structural performance



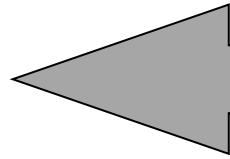
Main questions & challenges

- Is it possible to measure stresses with embedded magnetostrictive particles?
 - How to manufacture homogeneous specimen?
 - What is the effect of particle size?
 - How does the particle concentration change the sensitivity?
 - ...
 - How can the effect be used in a composite structure?



Approach

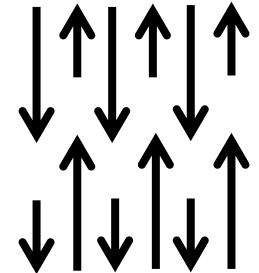
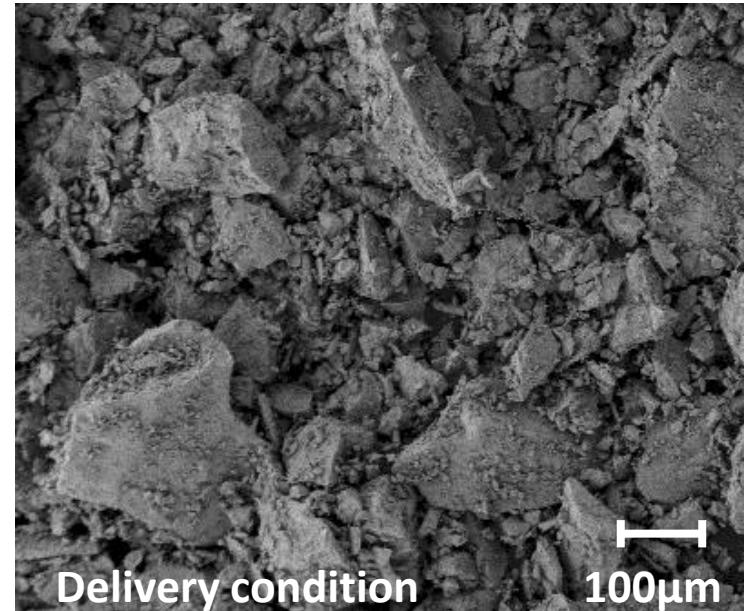
- Particle fractionation / characterization
- Particle dispersion
- Manufacturing of specimen
- Assessment of specimen
- Testing of specimen



Particles

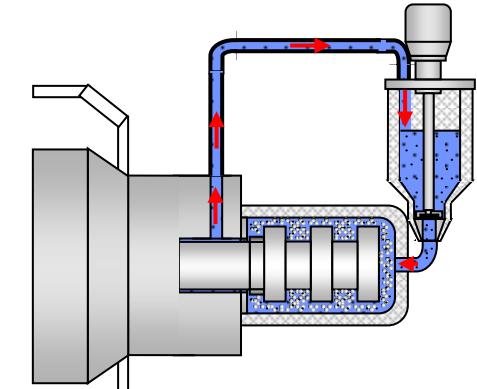
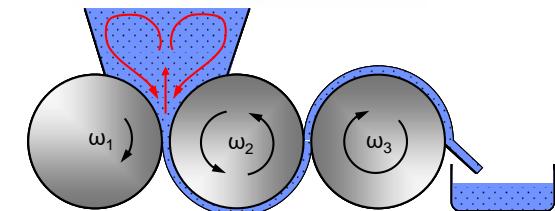
- Terfenol-D ($\text{Tb}_{0,3}\text{Dy}_{0,7}\text{Fe}_{1,92}$)
 - Terbium (42,3 wt%)
 - Dysprosium (40,0 wt%)
 - Iron (17,7 wt%)
- Selected properties:

• Density:	9,25 g/cm ³
• Young modulus:	25 - 35 GPa
• Tensile strength:	28 MPa
• Free strain:	800 - 1200 $\mu\text{m}/\text{m}$
• Ferrimagnetic	
- Delivered as Powder (ETREMA USA):
 - particle size: 0-300 μm
 - particle shape: irregular stochastic broken fragments (grinding process)



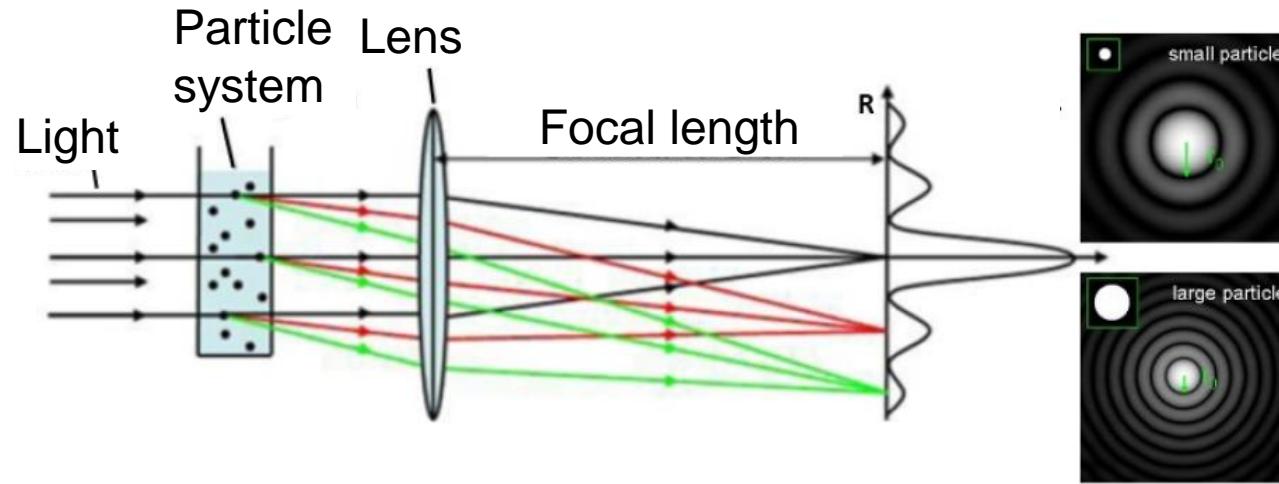
Particle fractionation

- Characterization and manipulation of particle sizes and particle size distributions
- Prerequisite to investigate the influence of the particle size
- Sieving of bulk particle powder:
 - Sieving tower: $0\text{-}300\mu\text{m} \rightarrow 0\text{-}100\mu\text{m}; 100\text{-}300\mu\text{m}$
 - Air jet sieve: $0\text{-}100\mu\text{m} \rightarrow 0\text{-}20\mu\text{m}; 20\text{-}100\mu\text{m}$
- Further processing of particle resin mixture:
 - Resin + $0\text{-}300\mu\text{m} \rightarrow$ master batch (32.25wt%)
 - Roll mill:
 - Gap mode $\rightarrow \sim 15\mu\text{m}$
 - Force mode $\rightarrow 0\text{-}15\mu\text{m}$
 - Bead mill $\rightarrow 0\text{-}10\mu\text{m}$



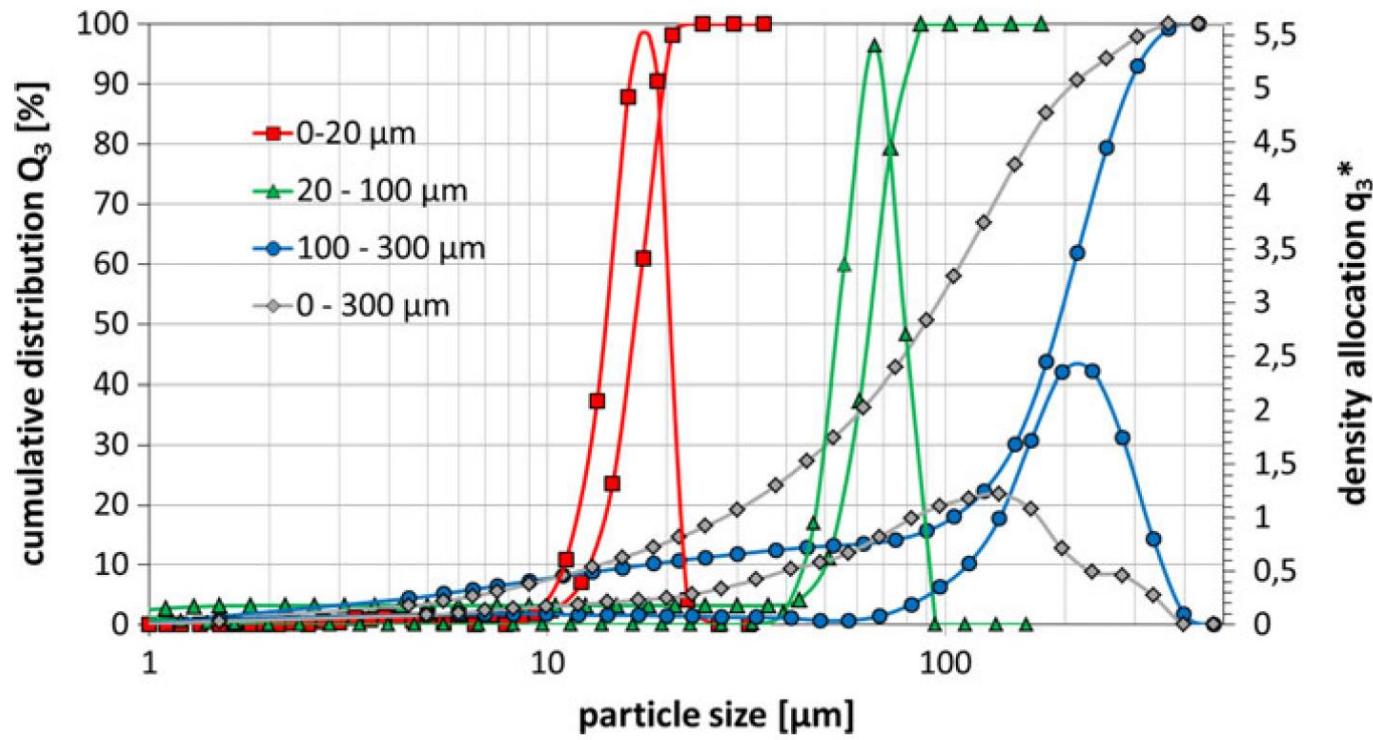
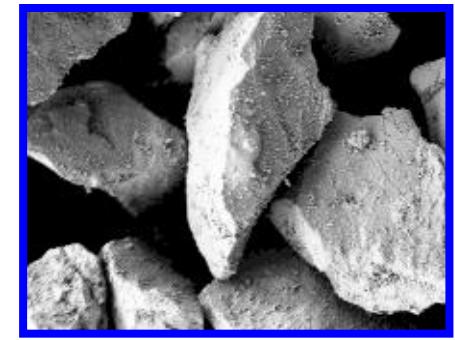
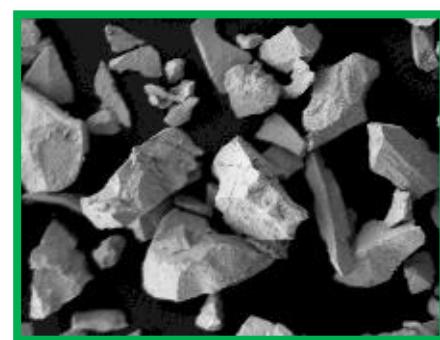
Particle fractionation

- Assessment via laser diffraction method (Laserspectrometer: Helios, Sympatec)
 - 100nm – 875µm



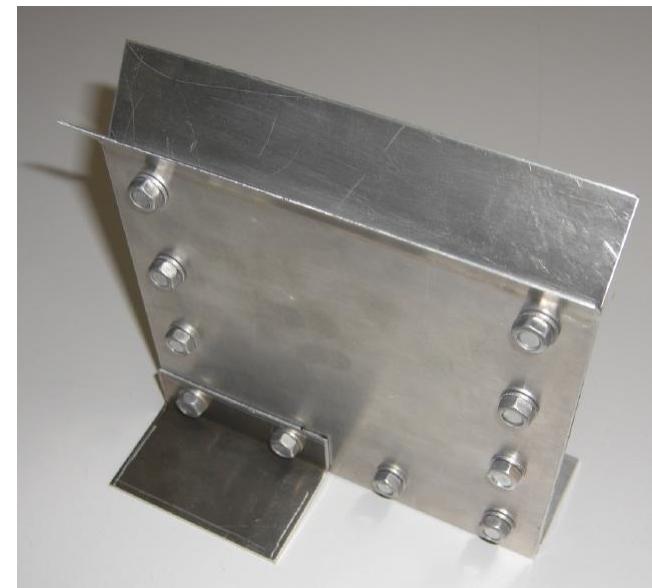
- Optical assessment using SEM
 - Directly possible after sieving
 - Separation of particles out of the processed master batch with acetone

Particle fractionation

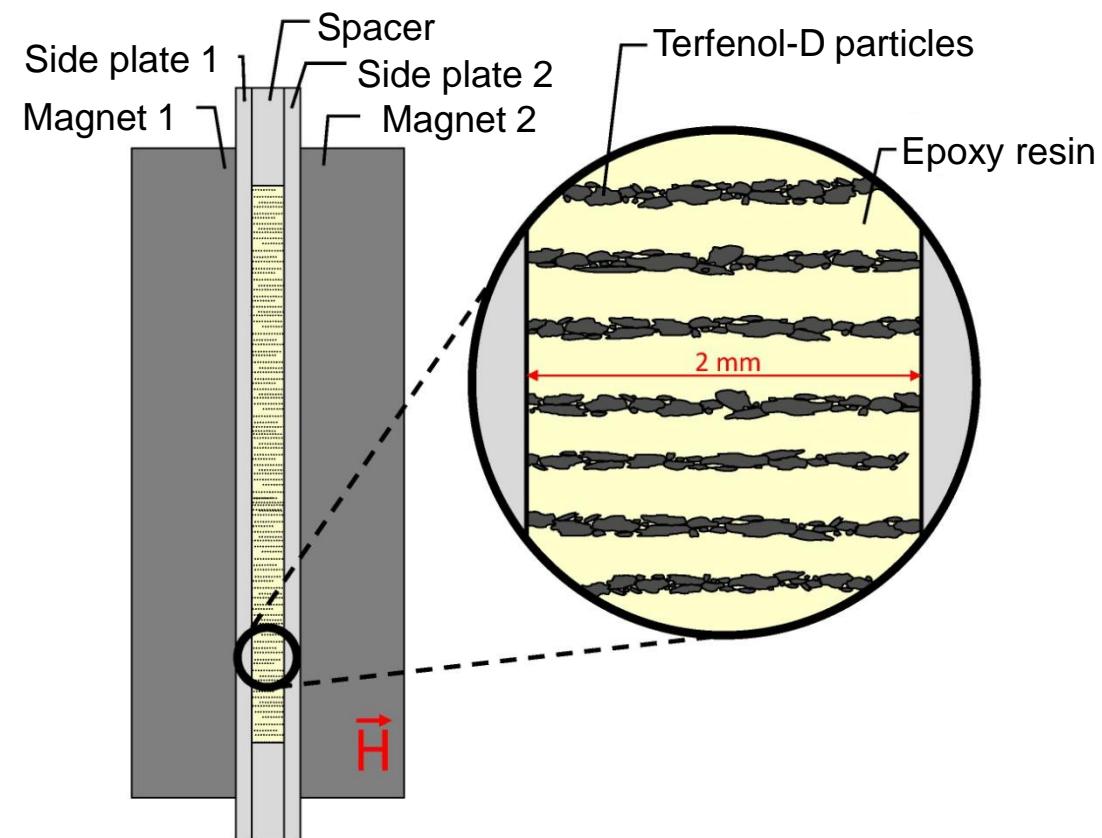
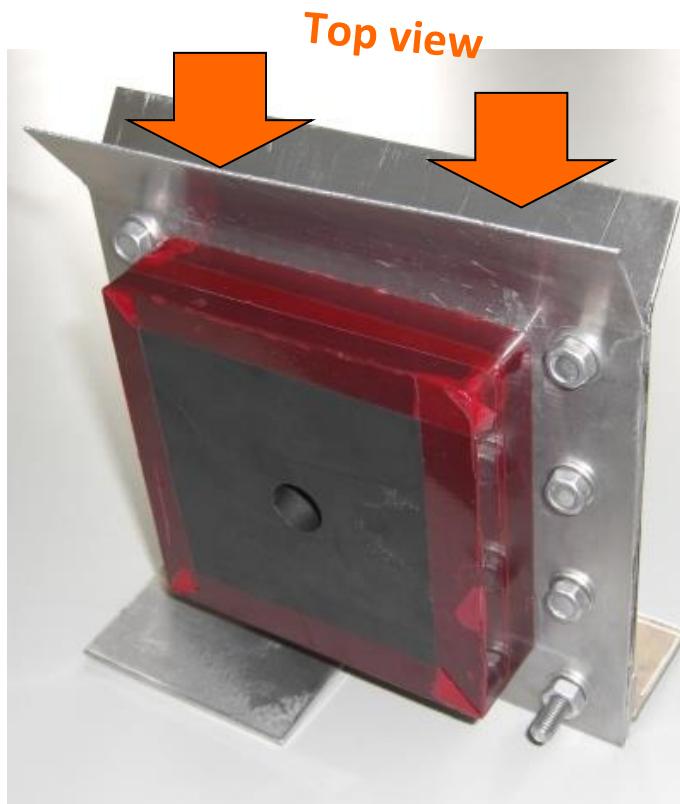


Particle dispersion

- 3 component Matrix (100/90/1; all by Huntsman):
 - Resin: Araldite LY 556
 - Hardener: Aradur HY 917
 - Accelerator: DY 070
- Particle sedimentation due to huge difference in density: 9.25g/cm^3 vs. 1.2g/cm^3
- Idea: combination of orientation and stabilization of the particles within a magnetic field



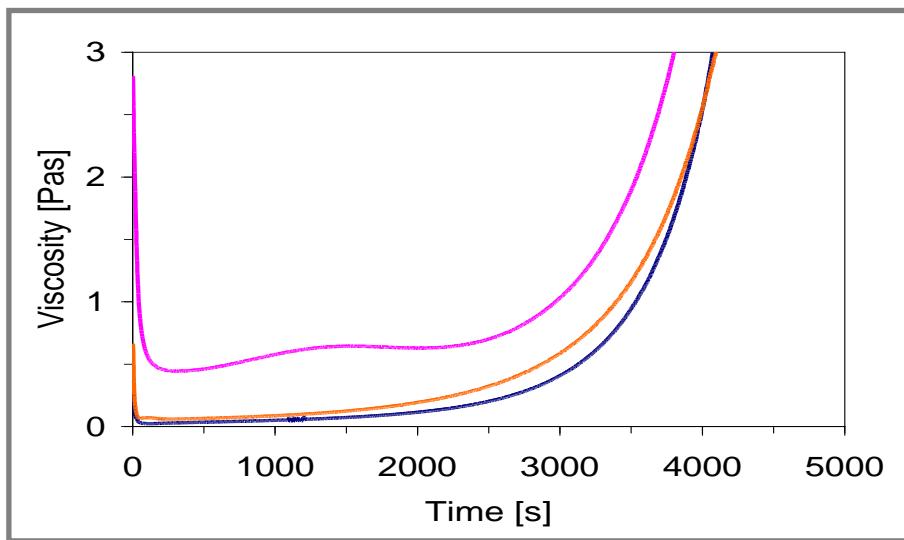
Manufacturing of Specimen



- Formation of particle chains
- Orientation of particles in the magnetic field
- Stabilization of the particles

Manufacturing of Specimen

- Field strength is crucial
- Too strong magnetic field leads to separation of the particles to both magnets
- Viscosity of the resin changes with time
 - Application of field at the right time
 - Electromagnet favorable



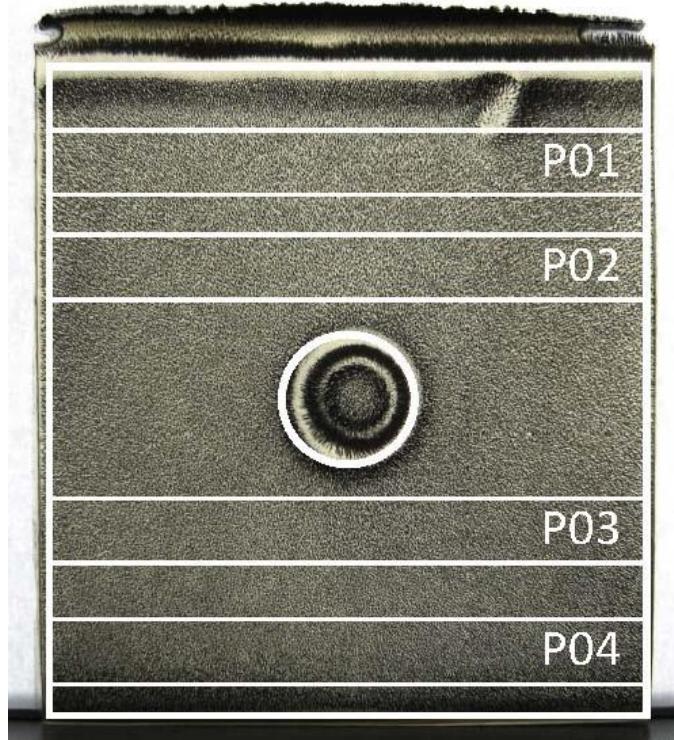
Specimen overview

	20wt%	5wt%
0-10µm	tbd.	tbd.
0-15µm	tbd.	tbd.
15µm	Optical	Optical
0-20µm	Optical / Mechanical	Optical / Mechanical
20-100µm	Optical / Mechanical	Optical / Mechanical
100-300µm	Optical	Optical
0-300µm	Optical / Mechanical	Optical



Optical assessment of specimen

- Specimen with different particle sizes (20 wt%)



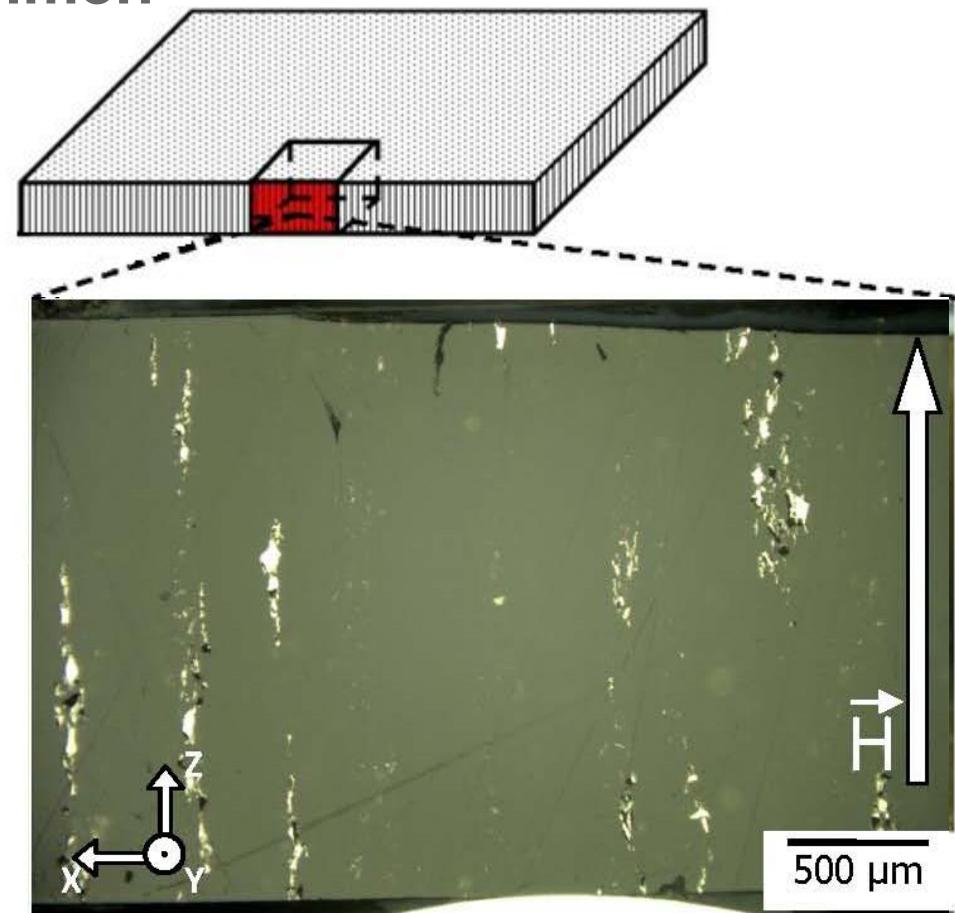
Particle size: 0 - 20 µm,
homogeneous, dense particle distribution



Particle size: 100 - 300 µm,
inhomogeneous particle distribution

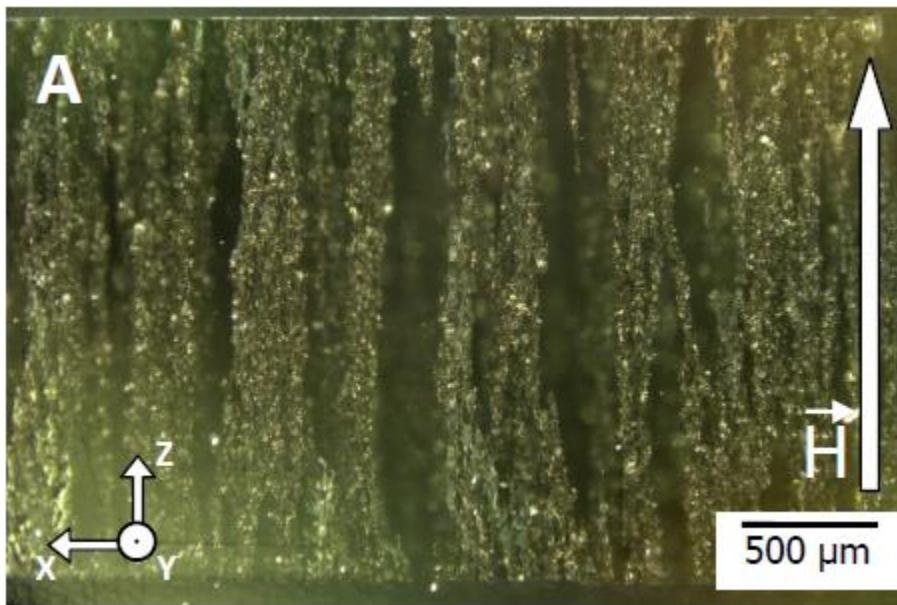
Optical assessment of specimen

- Optical Characterization of the particle distribution
- Light microscopy of a sample with 20 wt% particles of a size between 0-300 μm
- Beneficial use of polarization filter



Optical assessment of specimen

15µm, 20 wt%

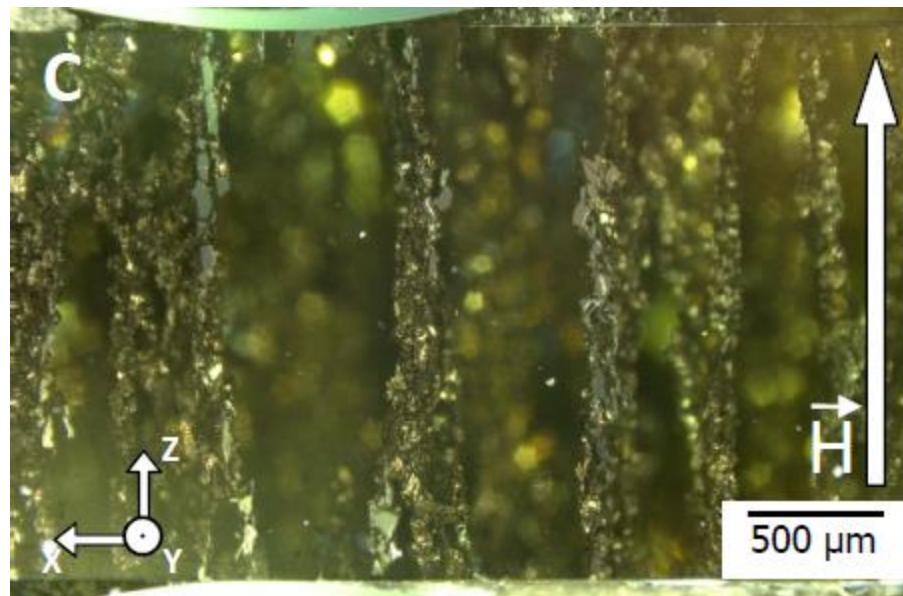


15µm, 5 wt%

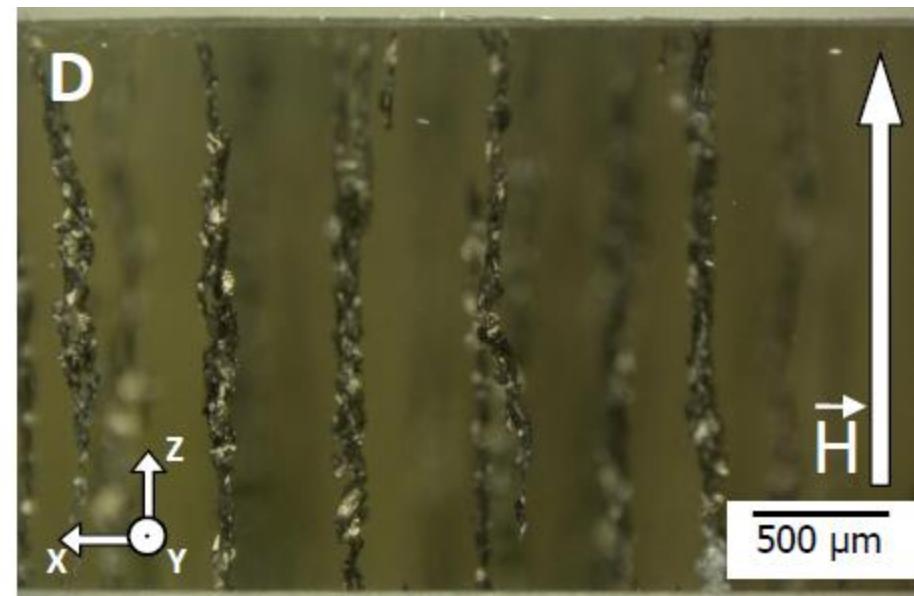


Optical assessment of specimen

0-20µm, 20 wt%

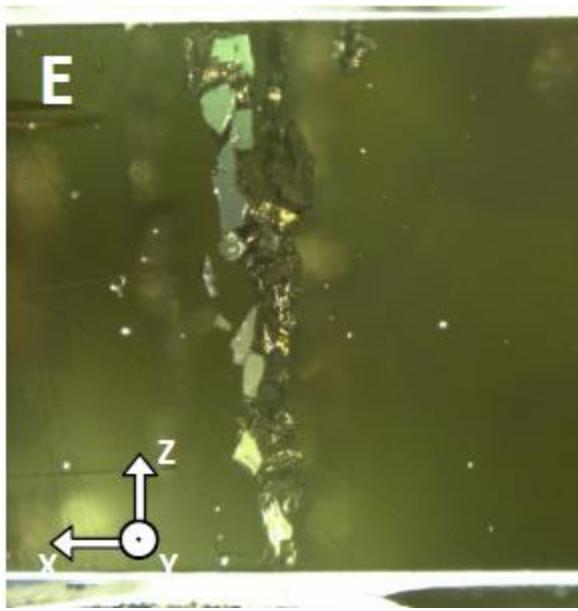


0-20µm, 5 wt%

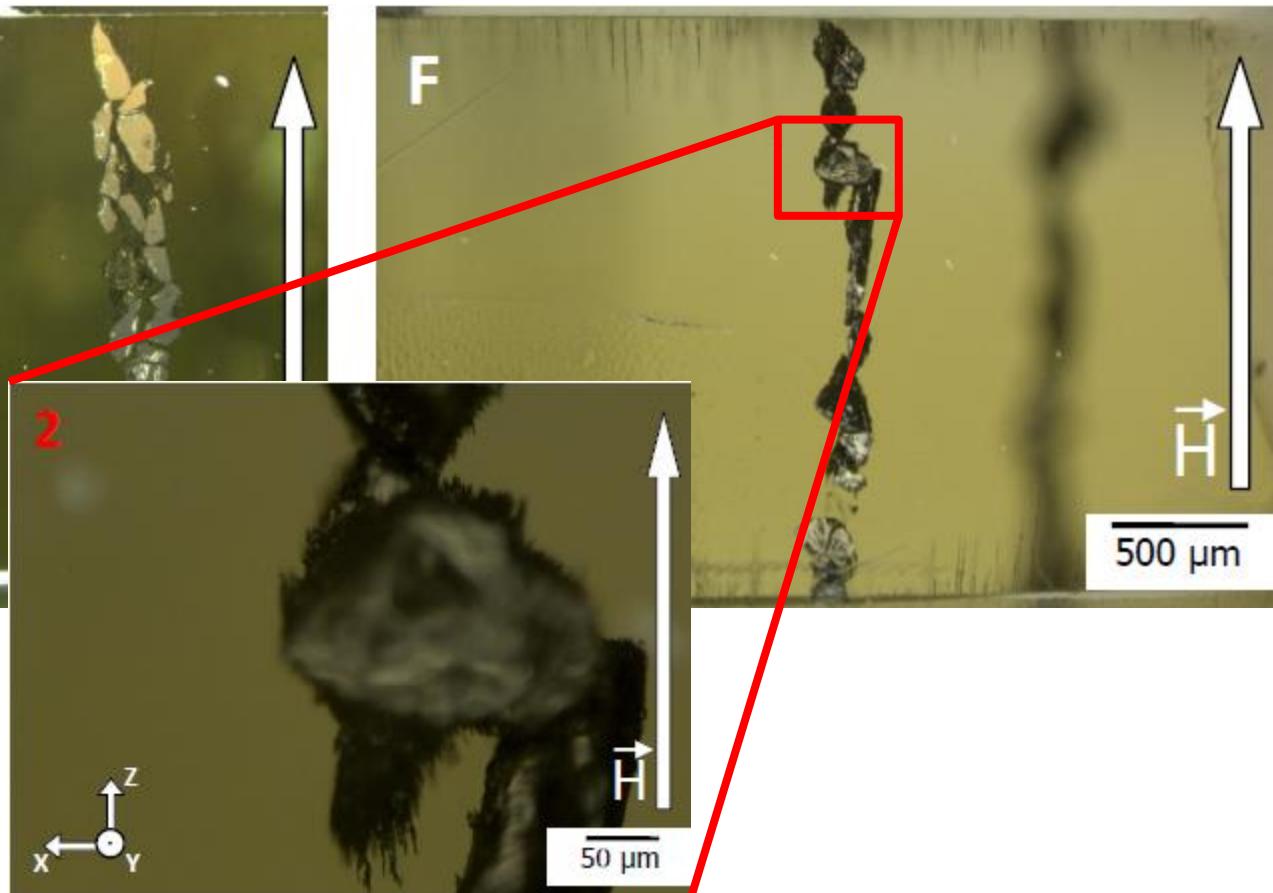


Optical assessment of specimen

100-300µm, 20 wt%

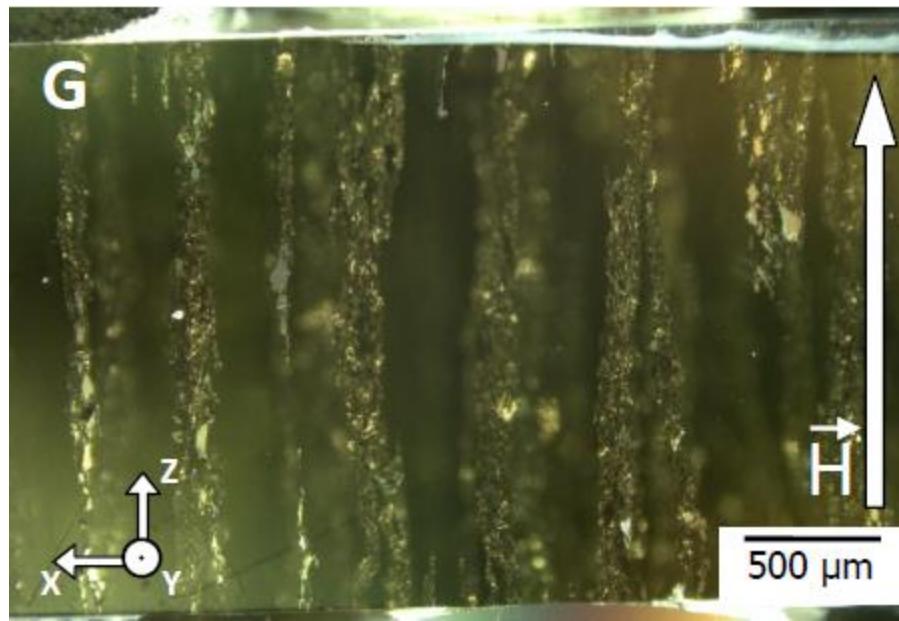


100-300µm, 5 wt%

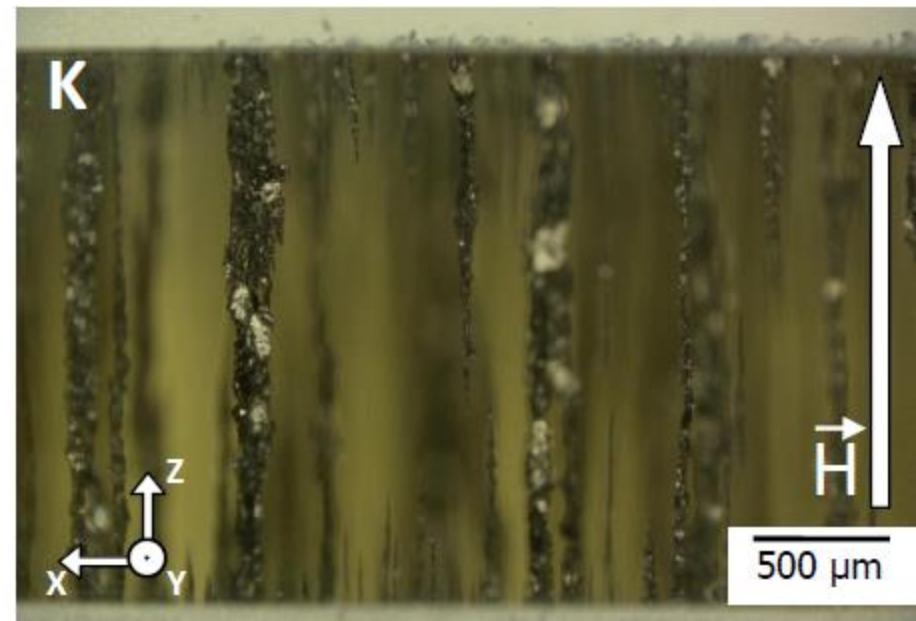


Optical assessment of specimen

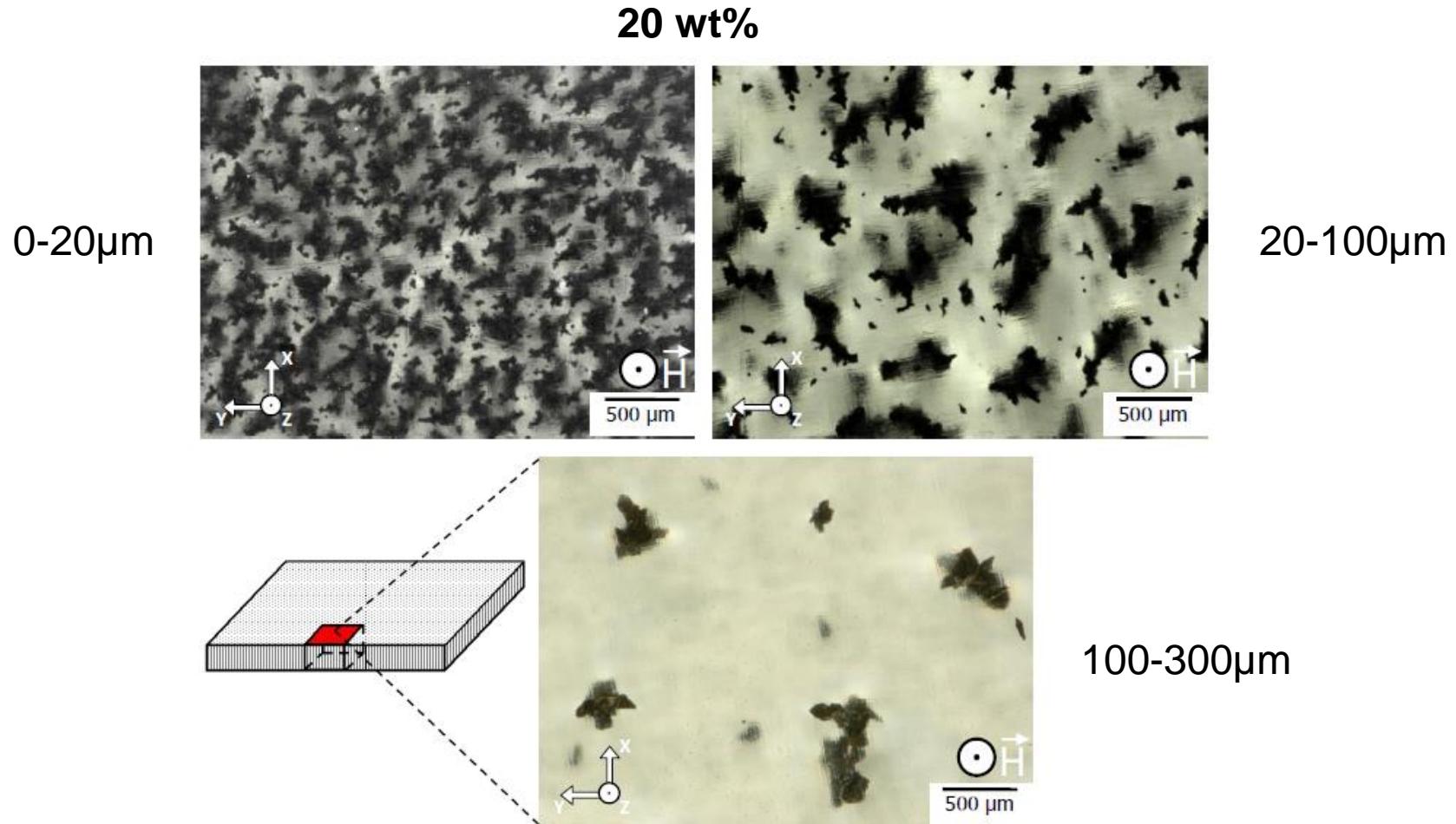
0-300µm, 20 wt%



0-300µm, 5 wt%

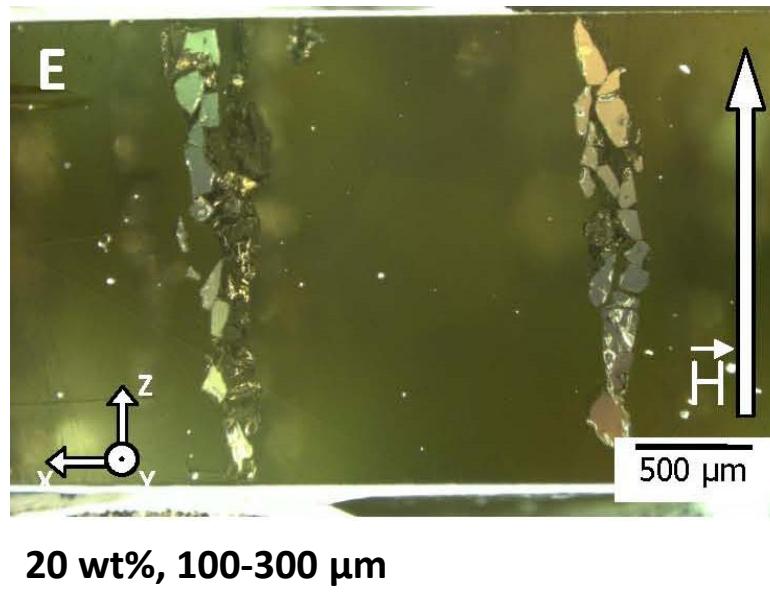
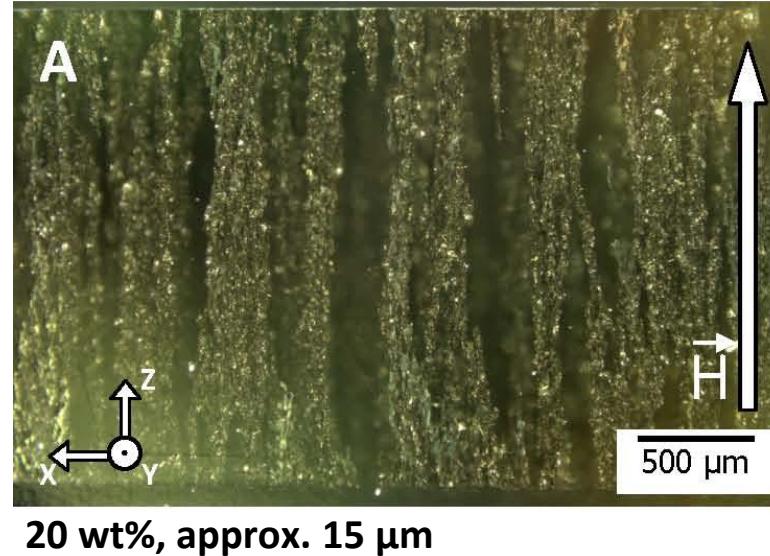


Optical assessment of specimen



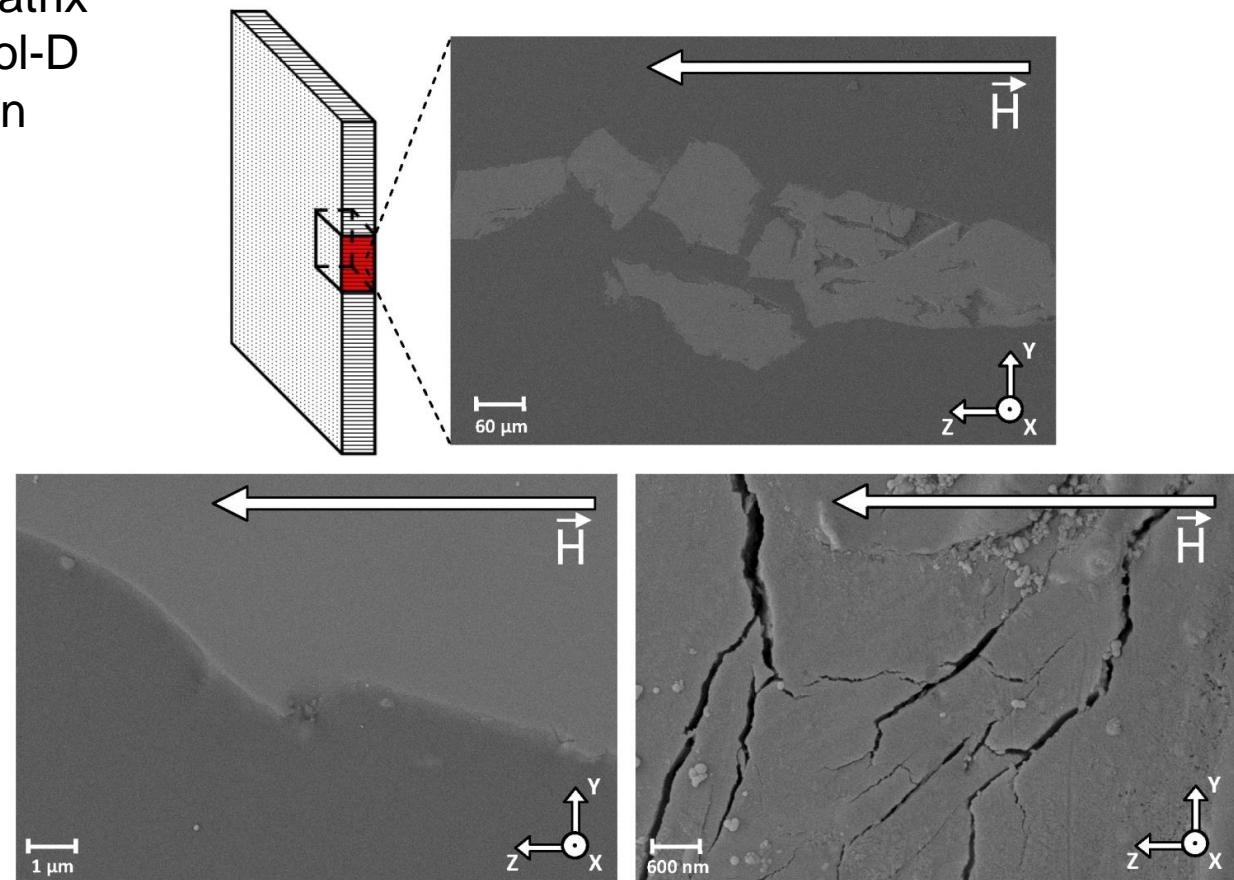
Conclusions of optical specimen assessment

- Small particles (<20µm):
 - Many particles / particle chain
 - High density of particles / particles chains
 - Fine structures
 - Chain to chain distance in the µm range→Homogeneous distribution
- Big particles(>100µm):
 - Few particles /particle chain
 - Low density of particles / particles chains
 - Coarse structures
 - Chain to chain distance in the mm range→Inhomogeneous distribution



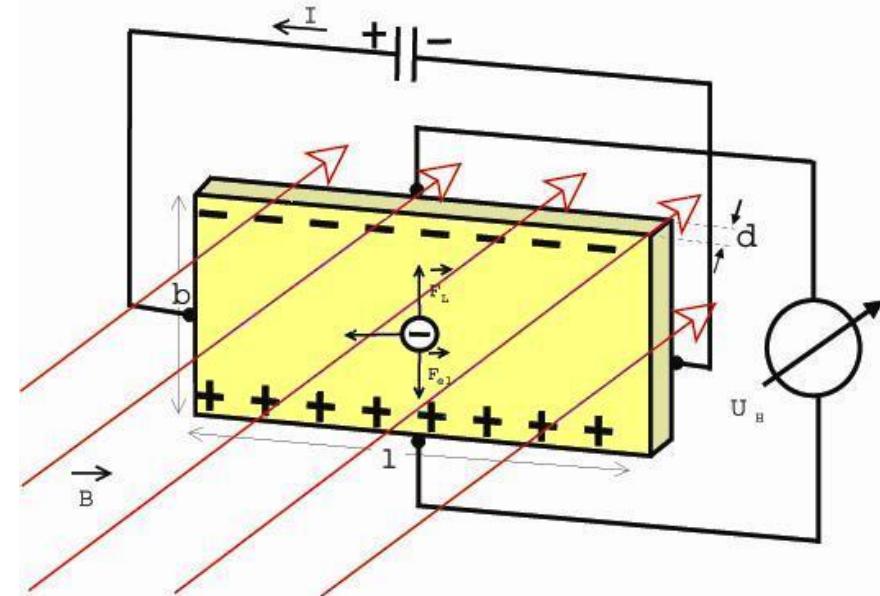
Particle-matrix adhesion

- SEM investigation of a specimen after loading (100-300 μm , 20 wt%):
 - Good adhesion to matrix
 - Cracks in the Terfenol-D
 - No cracks in the resin



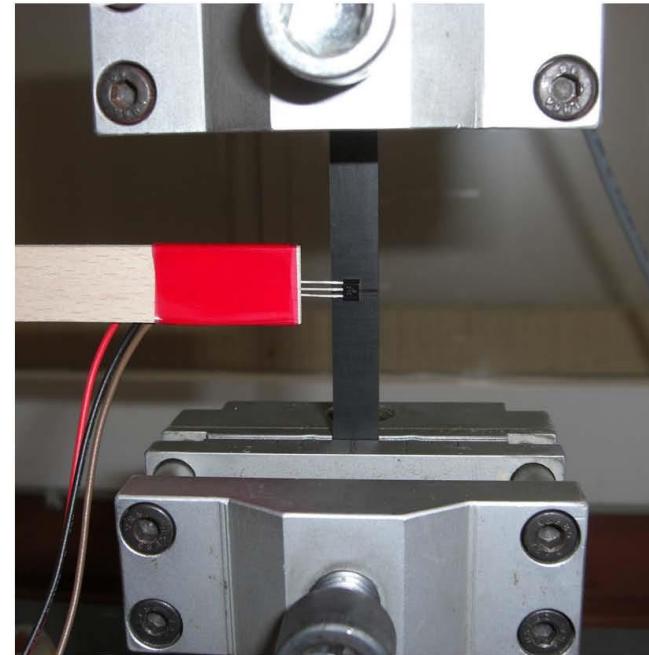
Test setup

- Equipment:
 - Universal testing machine
 - Measurement of traverse position
 - Measurement of force
 - Measurement of magnetic flux density via Hall-Sensor
 - Free moveable
 - Only relative measurements
 - Measurement range $\sim \pm 2\text{mT}$



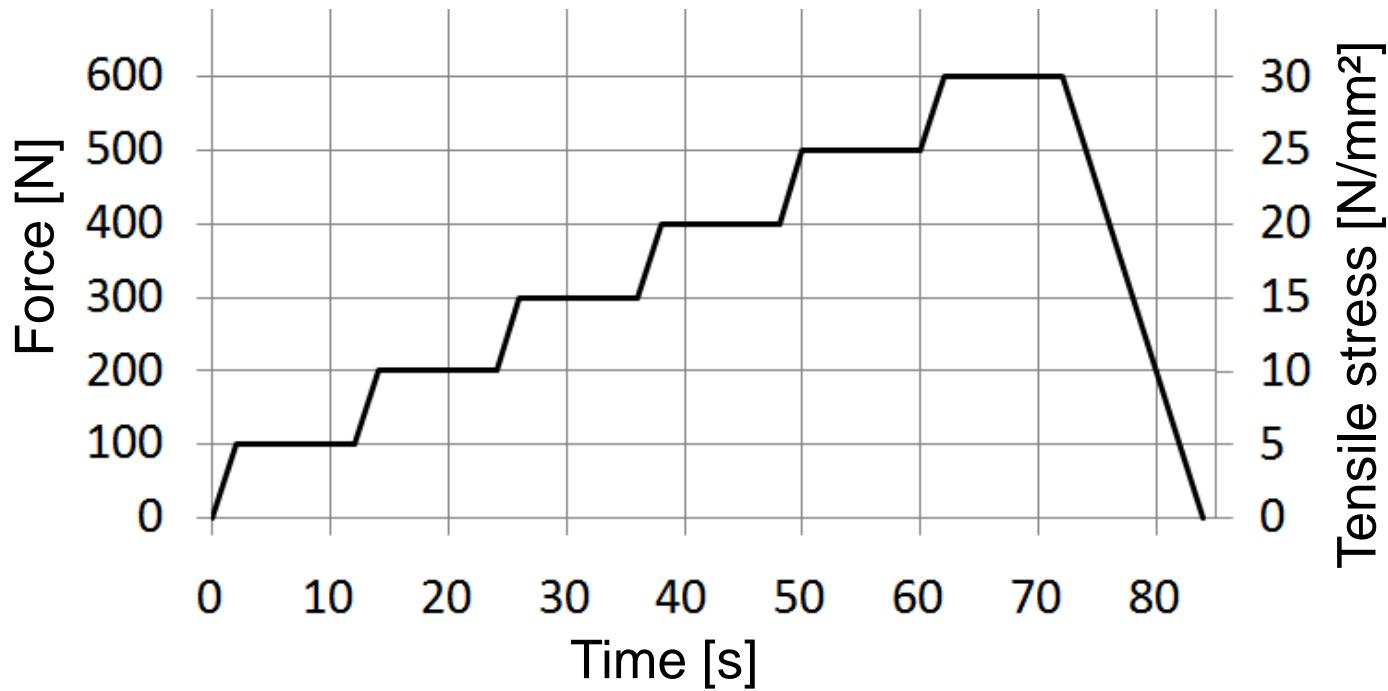
Test setup

- Measurement setup



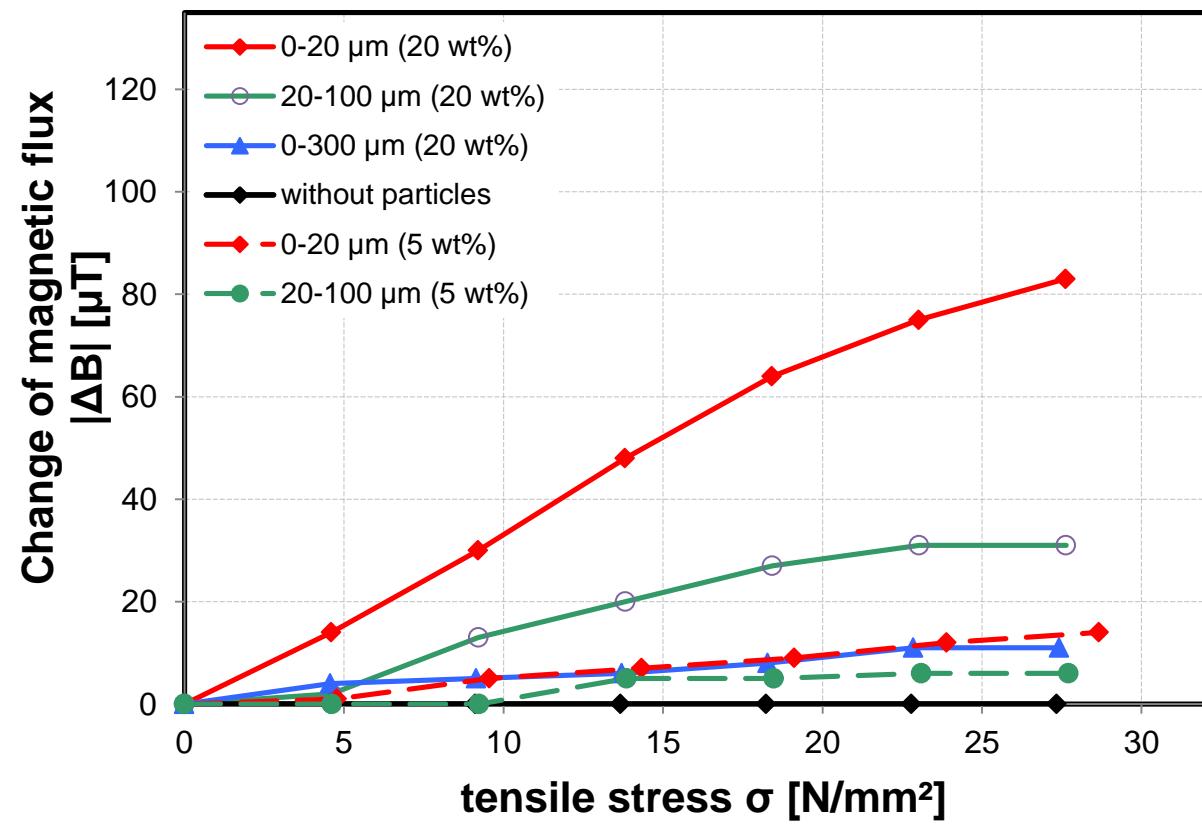
Test setup

- 6 force steps 100N each
- Loading speed: 2mm/min
- Stage dwell: 10s
- manual measurement of $|\Delta B|$ after 5s



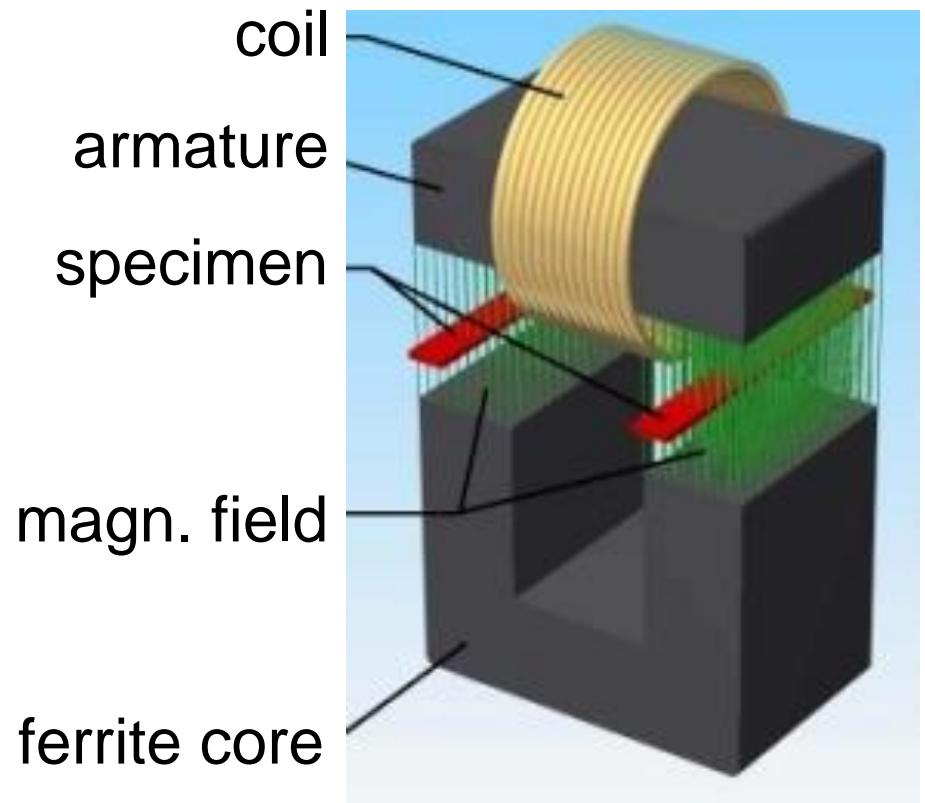
Experimental results

- Fairly small changes in flux density
- Clear dependency of stress
- Smaller particles show bigger effect
- Higher concentrations lead to higher sensitivity
- Stress almost reaches the tensile strength of Terfenol-D



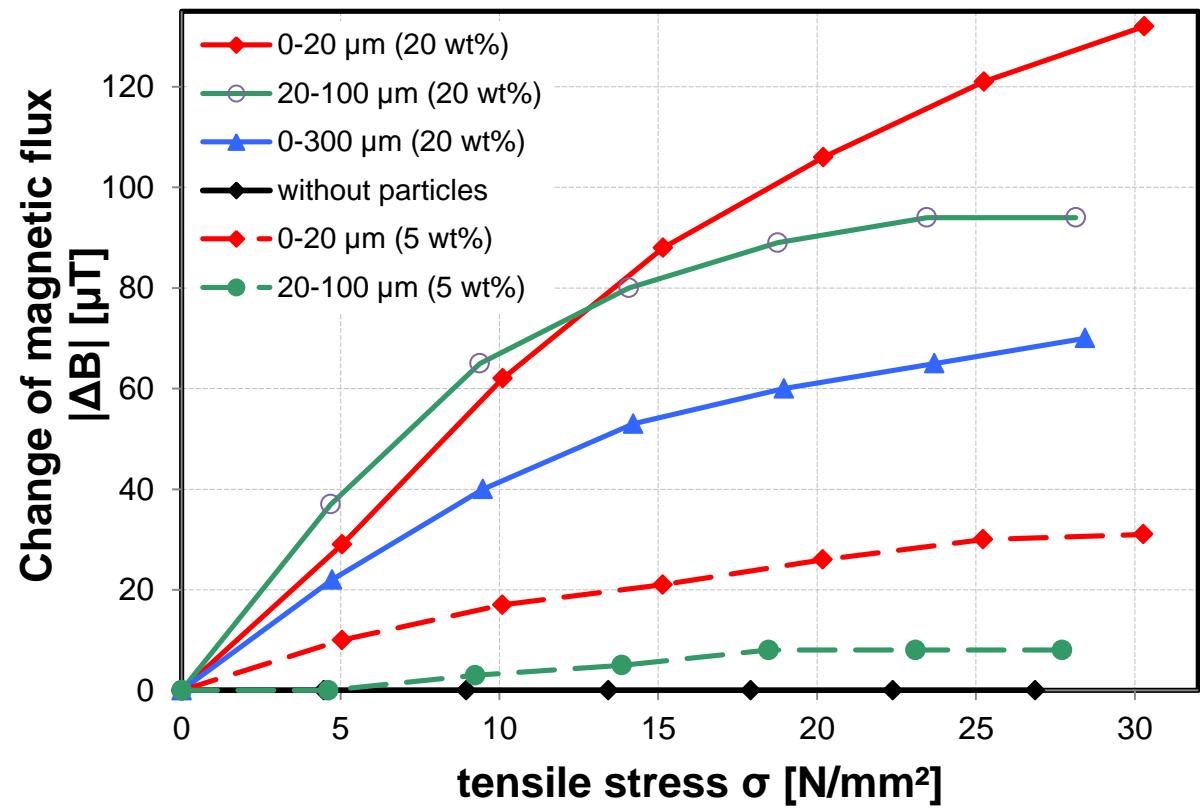
Experimental results

- 2nd magnetization
 - Duration: 30s
 - Magnetic flux density: 1.5T



Experimental results

- Magnetic response to external loading after 2nd magnetization:
 - Significant increase in sensitivity



Conclusions

- Change in flux density depends on:
 - Mechanical strain
 - Particle size
 - Particle content
 - Particle distribution
- Strong mutual influence
- Specimen with lower particle content showed lower changes in magnetic flux density
- Smaller particles seem to be favorable
 - Bigger signal
 - More uniform distribution
- Post magnetization leads to enhanced sensitivity



Future work

- Statistical confirmation
- analytical / numerical modeling
- Further reduction of particle size → nano?
- Impact of mechanical and rheological properties (e.g. resin viscosity)
- Tuning of process parameters
- Manufacturing of composite specimens
 - Challenge: orientation and stabilization of particles in big or complex parts, definition of zero state → residual stresses
 - Impact on mechanical properties of the laminate
- Improvement of test and manufacturing equipment
- Other sensor materials (lower density, better availability)



Thanks for your attention.

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more information:

www.leichtbau.dlr.de/
www.DLR.de/FA



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