Manufacture and Thermomechanical Characterisation of Wet Filament Wound C/C-SiC Composites

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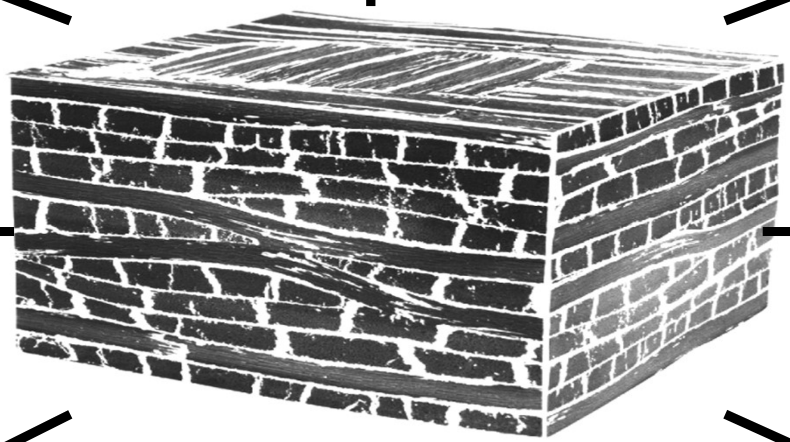
Overview

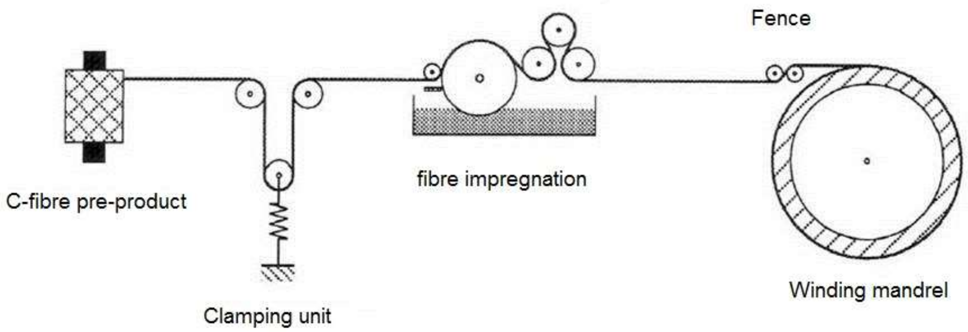
- **Motivation: general properties of C/C-SiC**
- **Preform manufacture: wet filament winding and LSI-processing**
- **Microstructure of C/C-SiC in dependence of winding angle**
- **Thermomechanical testing: sample preparation and mechanical testing via Indutherm**
- **Results of mechanical testing**
- **Fracture surfaces of tested specimens**
- **Conclusions**
- **Outlook and acknowledgement**

Motivation: General Properties of C/C-SiC Materials

- High thermal shock stability
- Damage tolerance
- Internal oxidation protection
- Good wear and corrosion resistance
- Variable mass fractions of SiC and C
- Low density (ca. 2 g/cm³)
- Low porosity
- Short manufacturing times



Preform Manufacture - Wet Filament Winding of Water-based Phenolic Resin



Warm Pressing for manufacture of plate material

Curing of flattened green bodies in an autoclave with gas pressure for manufacture of plates

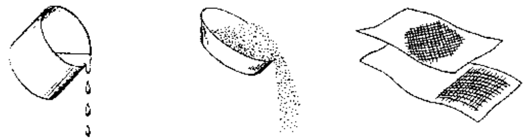
Pressure less curing on mandrel in oven for manufacture of tubes

Raw materials and equipment:

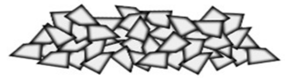
- C-fibre T800 12K and precursor MF43 (water-based phenolic resin) with high C-yield
- Filament winding machine controlling winding speed and angle
- Aluminium mandrel equipped with Teflon tape (Ø = 335 mm, h = 220 mm))
- IR-lamp and ventilation for support of evaporation of solvent
- Cutting of wet CFRP-tube and curing of flattened green body via autoclave

Liquid Silicon Infiltration Process (LSI)

Precursor **Additives**
Fibres



Silicon-Granulate



CFRP (SHAPING)
RTM-, Autoclave-, Filament-Winding-, Press-Technique
T < 250 °C

→

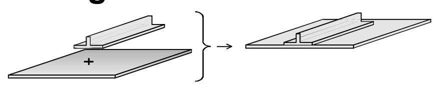
PYROLYSIS
Matrix Shrinkage
T > 900 °C
Inert Gas

→

SILICONIZATION
Si + C → SiC (Matrix)
T > 1420 °C
Vacuum

Intermediate Machining

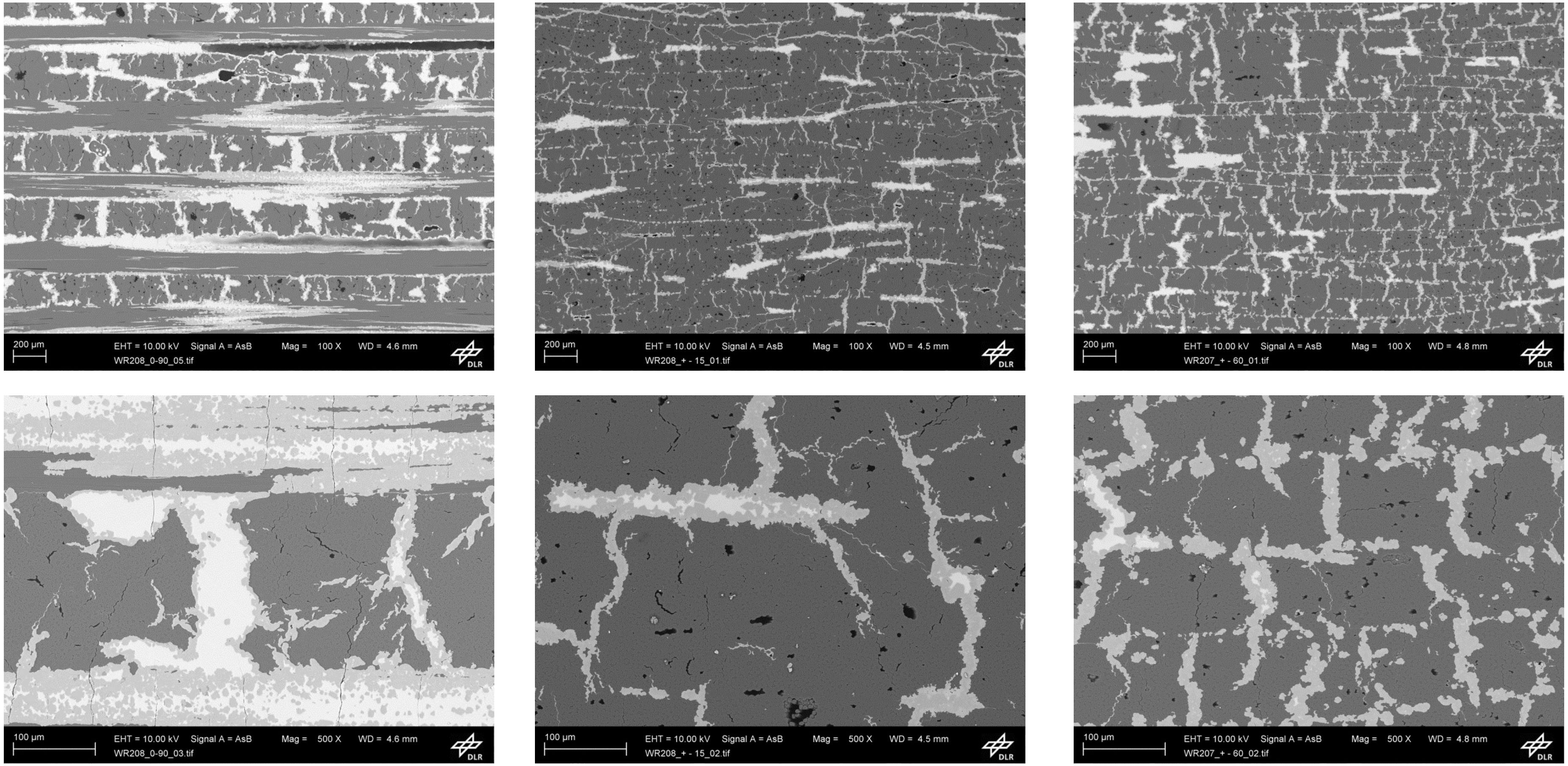
Joining
Fixing C/C-Parts with Paste



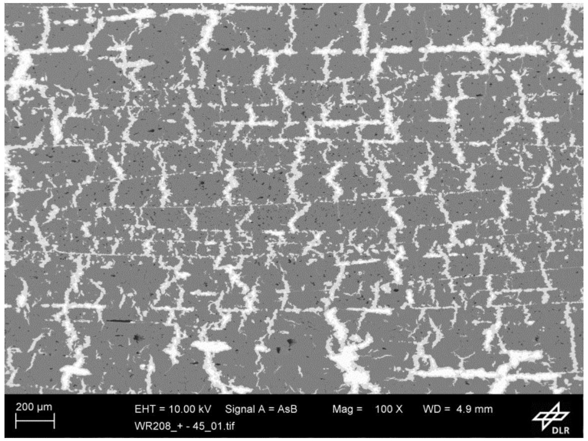
In-process coating (SiCralee)

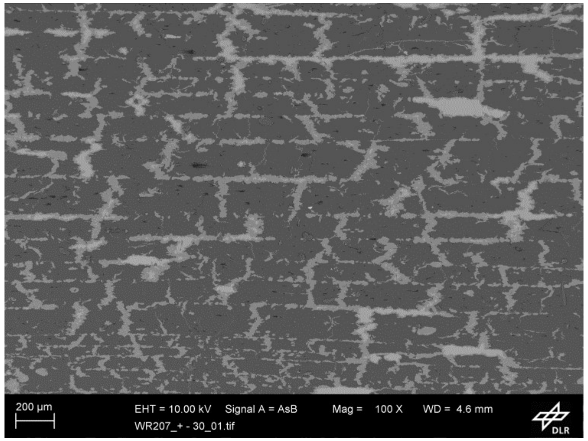
Finishing
Machining with Diamond Tools

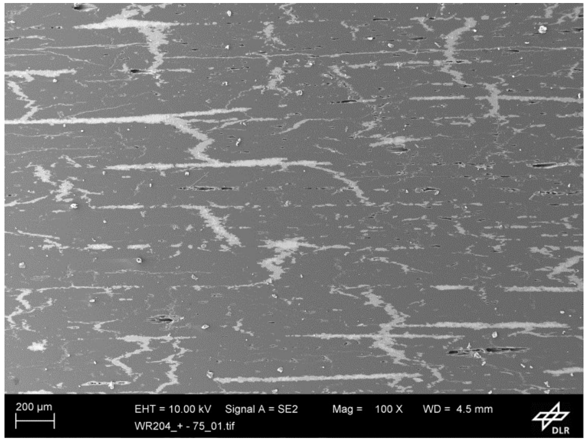
Microstructure of C/C-SiC w.r.t. Fibre Orientation (0°/90°(l.), ±15°(m.), ±30°(r.))

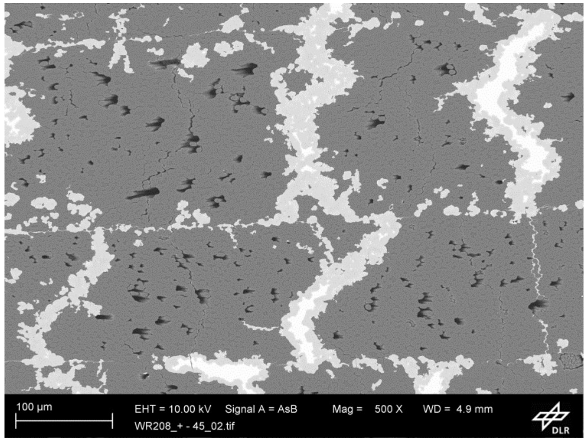


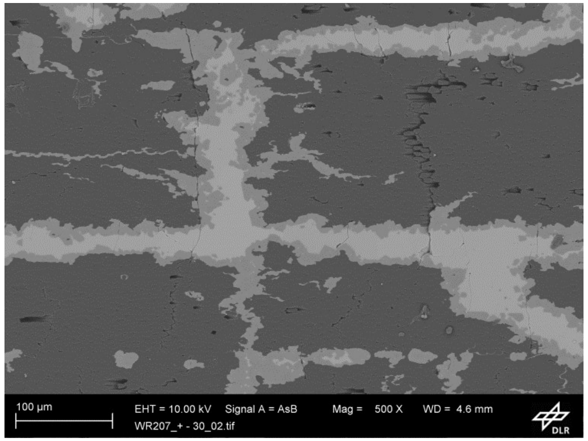
Microstructure of C/C-SiC w.r.t. Fibre Orientation (±45°(l.), ±60°(m.), ±75°(r.))

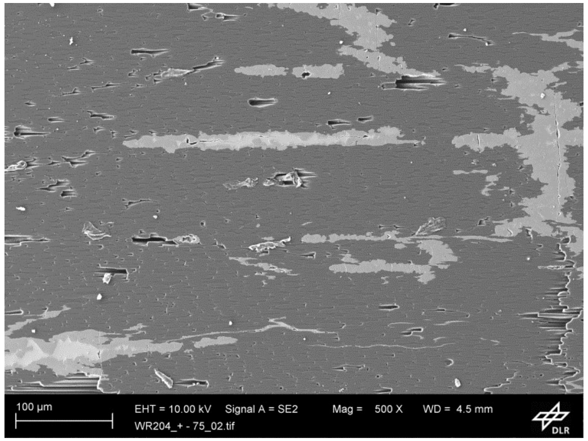




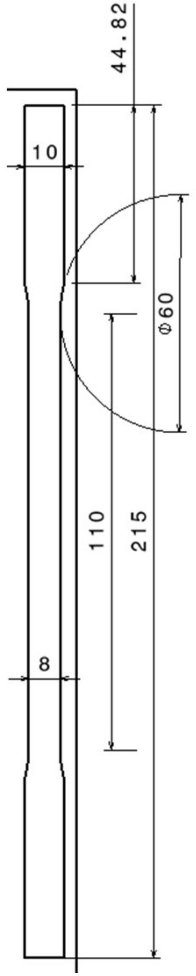


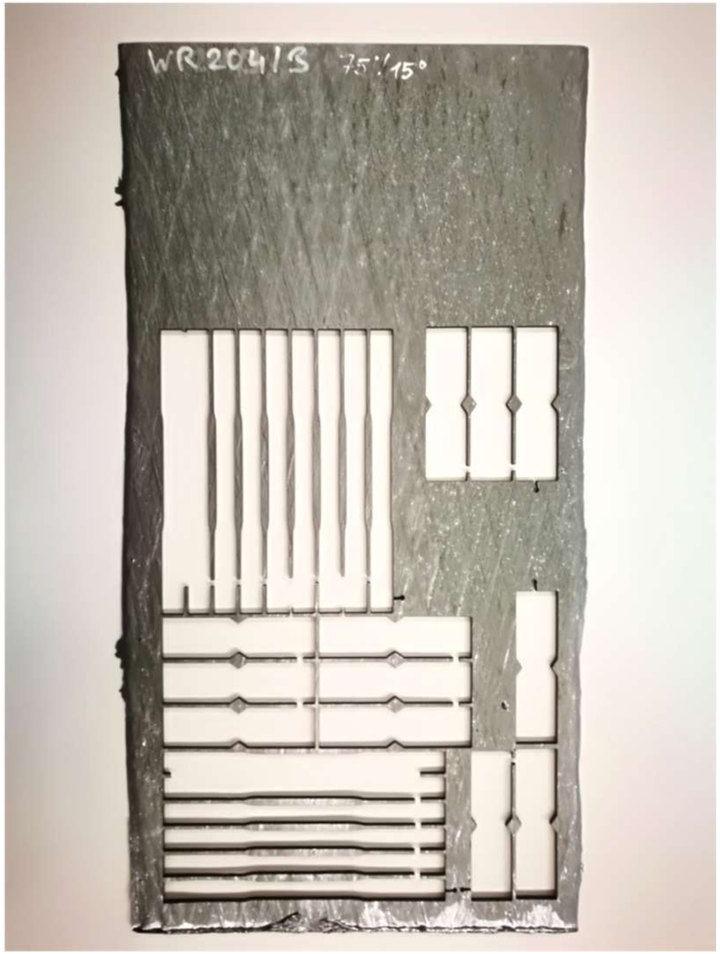


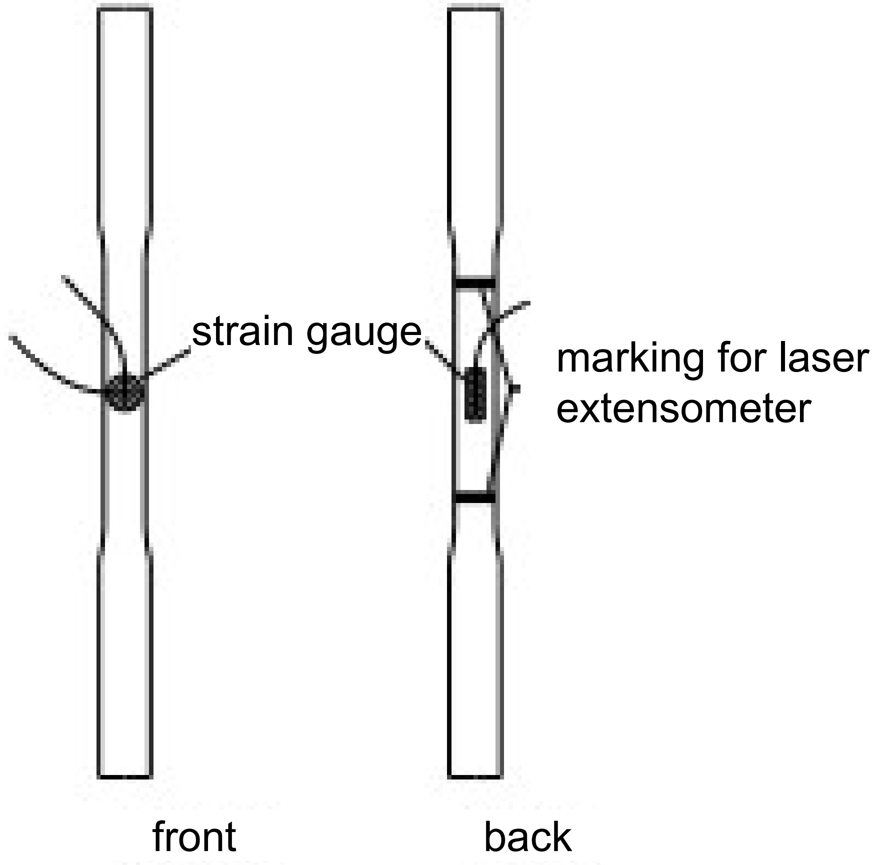




Procedure for Specimen Preparation via Jet-cutting from C/C-SiC Plates







Tensile Testing under Static Loading

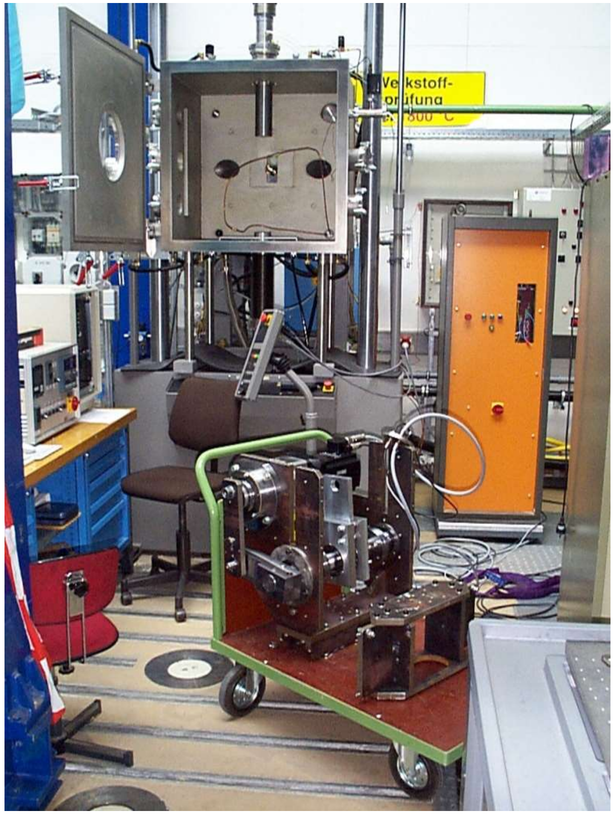
- Tensile testing on universal testing machine (Zwick UTS) using strain gauges for measure of elongation at room temperature

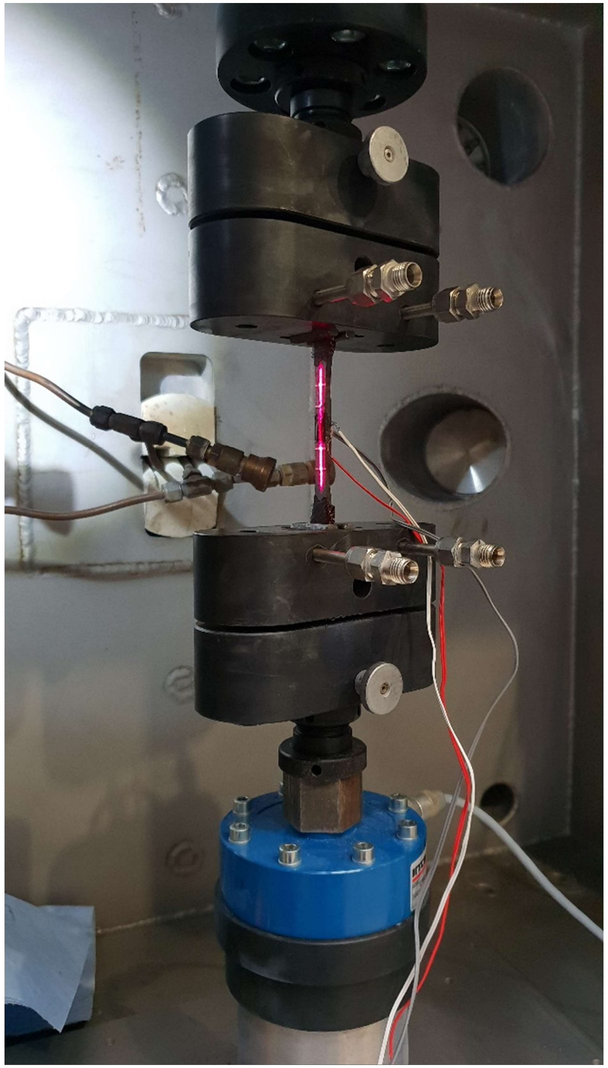
- Tensile testing on universal testing machine (Indutherm) using strain gauges to measure of elongation at room temperature and to compare results to a laser extensometer, which will be used for measurement at high temperatures

- Tensile testing on universal testing machine (Induthern) using a laser extensometer, which will be used for measurement of elongation at high temperatures

Indutherm: RT-set-up

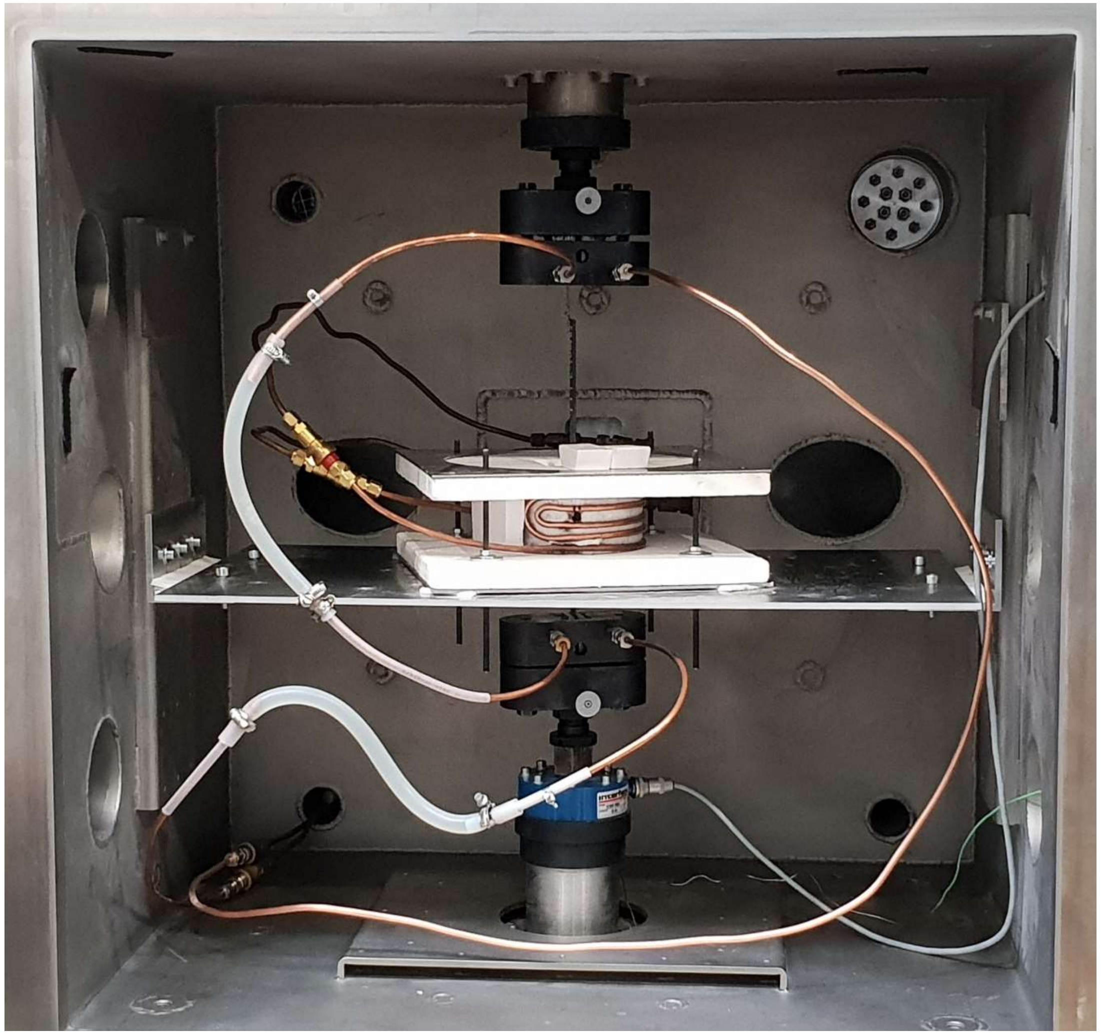
- Room temperature and ambient pressure
- C/C-SiC flat specimens
- Strain gauges and laser extensometer for comparison of fracture strain
- Fixation via tensioning wedges in order to avoid additional side forces
- Training of personel to machine and marking possibilities





Indutherm: HT-set-up

- Reduced N_2-pressure in chamber with less than1 mbar
- C/C-SiC flat specimens with cold clamping and distinct marking for measure of strain at HT via laser extensometer
- Selection of marking material is decisive for success (reactivity, radiation, etc.)
- Isolation (alumina) in order to shield clamping devices from radiation
- High temperature via inductive heating at high heating rates (water-cooled copper tubes)
- Fast cooling to RT for multiple test runs within one day



Mechanical properties, density and open porosity of C/C-SiC composite plates at various fibre orientations

Fibre orientation [°]	0/90	±15	±30	±45	±60	±75
Plate number	WR208	WR204	WR206	WR208	WR206	WR204
Open porosity (CFRP) [vol.-%]	6.22	3.10	4.61	6.22	4.61	3.10
Open porosity (C/C) [vol.-%]	15.7	15.0	14.6	15.7	14.6	15.0
Si-uptake [mass-%]	40.2	34.5	38.2	40.2	38.2	34.5
Tensile strength [MPa]	n.d.	264.9±24.8	172.1±20.1	58.7±24.7	35.2±2.6	15.7±0.4
Tensile modulus [GPa]	n.d.	137.6±9.9	70.0±8.4	26.9±7.8	19.0±3.0	22.0±4.7
Fracture strain [%]	n.d.	0.21±0.02	0.39±0.05	0.33±0.14	0.55±0.11	0.12±0.07
Density C/C-SiC) [g/cm³]	2.02	1.86	2.07	2.02	2.07	1.86
Open porosity (C/C-SiC) [vol.-%]	2.74	2.10	1.23	2.74	1.23	2.10

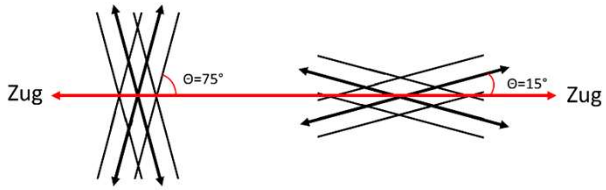
Mechanical properties, density and open porosity of C/C-SiC composite plates at various fibre orientations at 1300°C and at room temperature (RT)

Fibre orientation [°]	15	30	45	60	75
Plate number	WR204	WR206	WR208	WR206	WR204
Tensile strength [MPa]	294.2±12.2	238.1±7.3	106.2±3.9	n.d.	n.d.
Tensile modulus [GPa]	140.4±46.4	96.3±4.3	26.6±3.4	n.d.	n.d.
Fracture strain [%]	0.18±0.01	0.37±0.03	0.75±0.07	n.d.	n.d.
Tensile strength [MPa]	264.9±24.8	172.1±20.1	58.7±24.7	35.2±2.6	15.7±0.4
Tensile modulus [GPa]	137.6±9.9	70.0±8.4	26.9±7.8	19.0±3.0	22.0±4.7
Fracture strain [%]	0.21±0.02	0.39±0.05	0.33±0.14	0.55±0.11	0.12±0.07

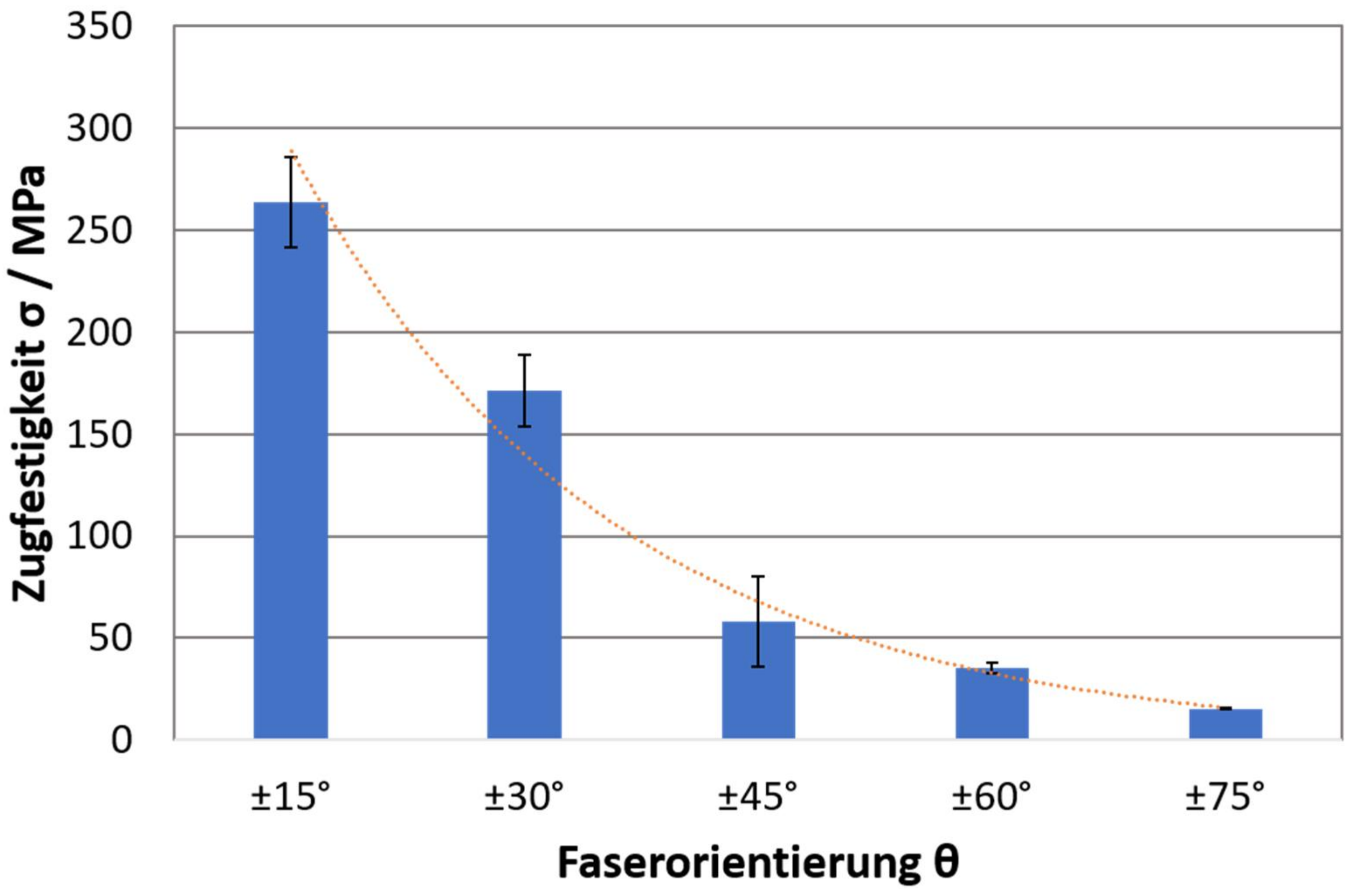
→ higher tensile strength at 1300°C than at RT, tensile modulus and fracture strain unchanged
→ C/C-SiC has excellent high temperature strength for high temperature applications

Tensile Strength @ Room Temperature

- Increaing angle of fibre orientation
  - ➢Lower tensile strength



- Relatively high standard deviation at ± 45° fibre orientation

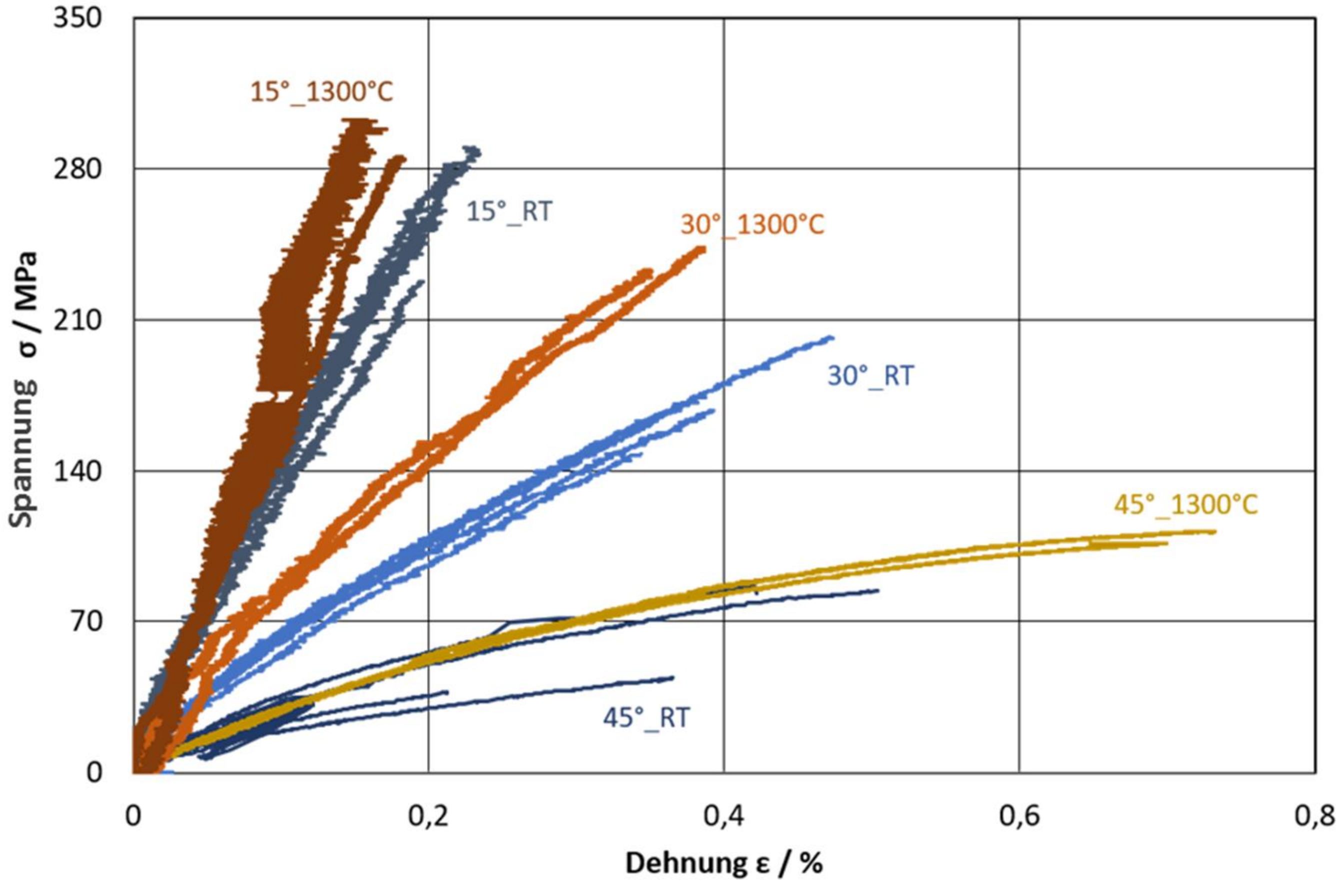


Tensile Strength @ Temperature: HT - RT

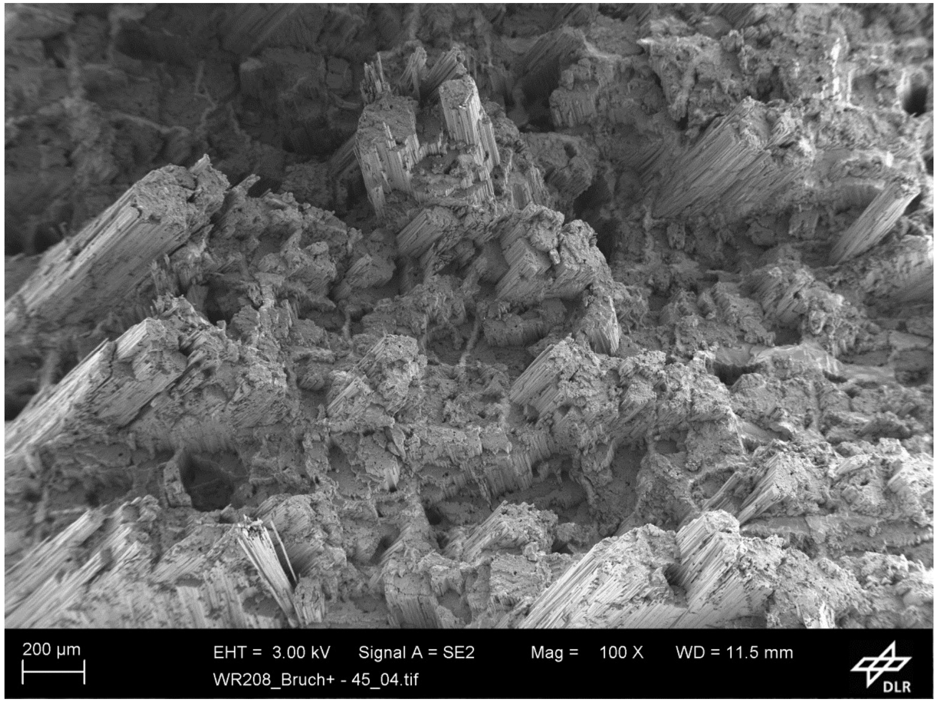
- **± 15°**
  ca. 10% higher tensile strength
  ca. 15% lower fracture strain

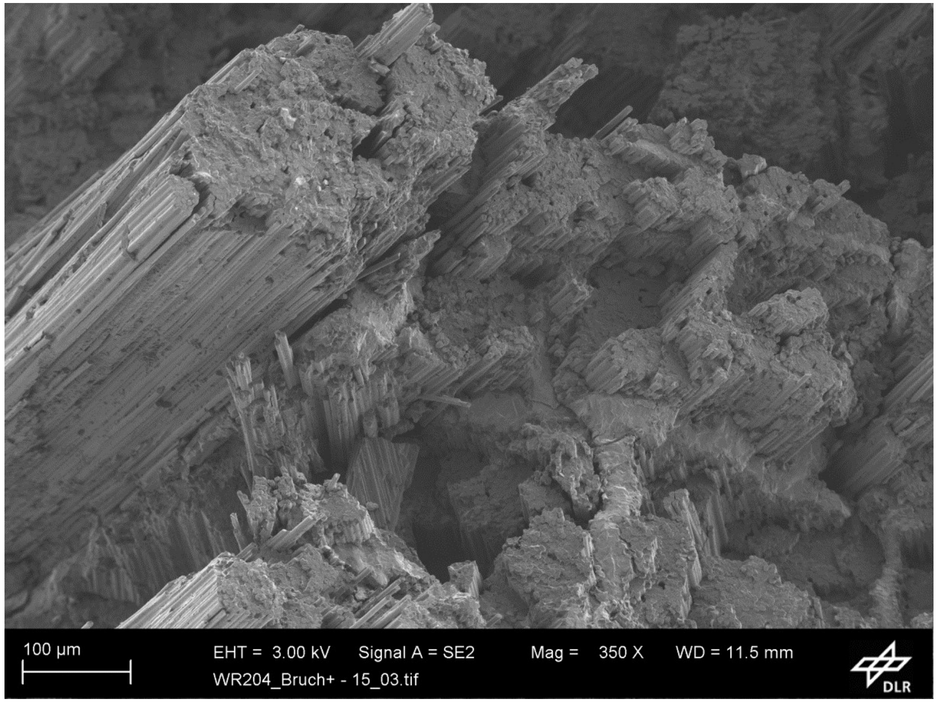
- **± 30°**
  ca. 28% higher tensile strength
  ca. 5% lower fracture strain

- **± 45°**
  ca. 47% higher tensile strength
  ca. 54% higher fracture strain



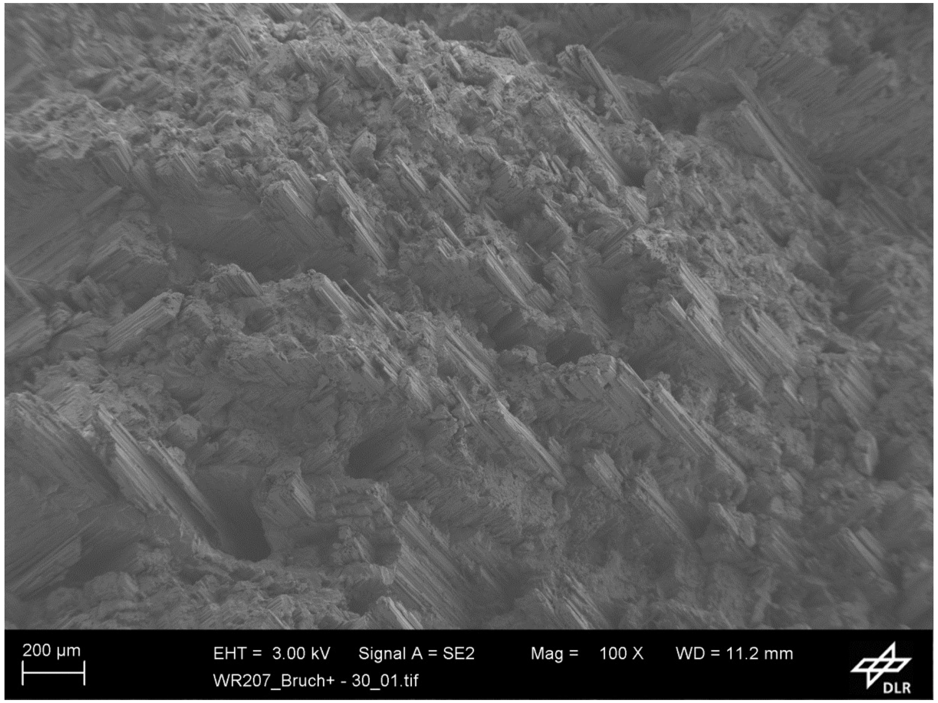
Fracture Surface with Fibre Orientation to Loading Direction (±15°C)

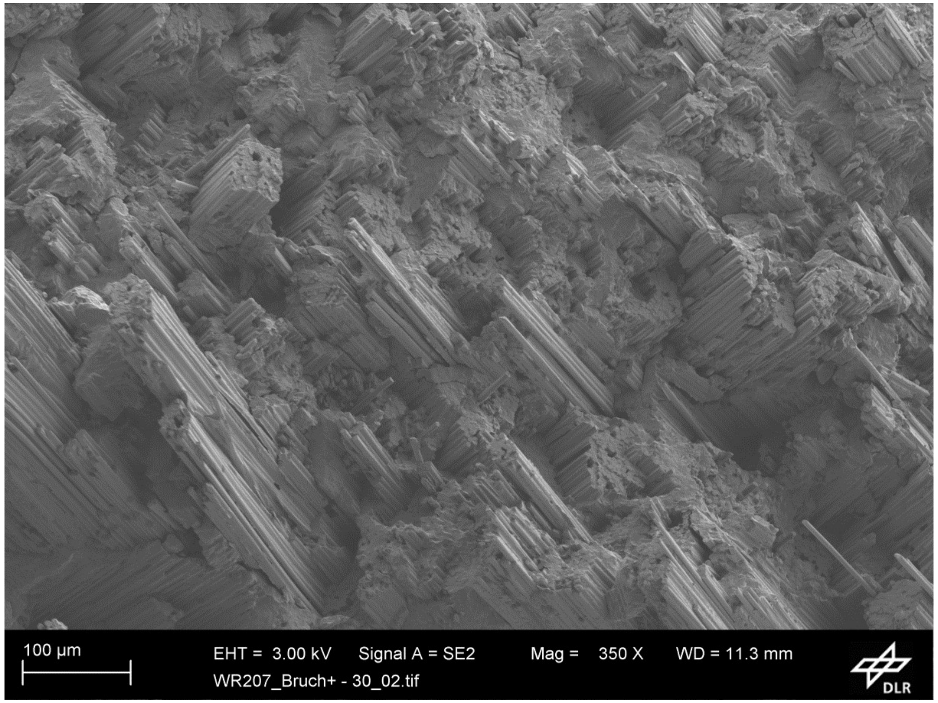




→ rather high fibre pull-out

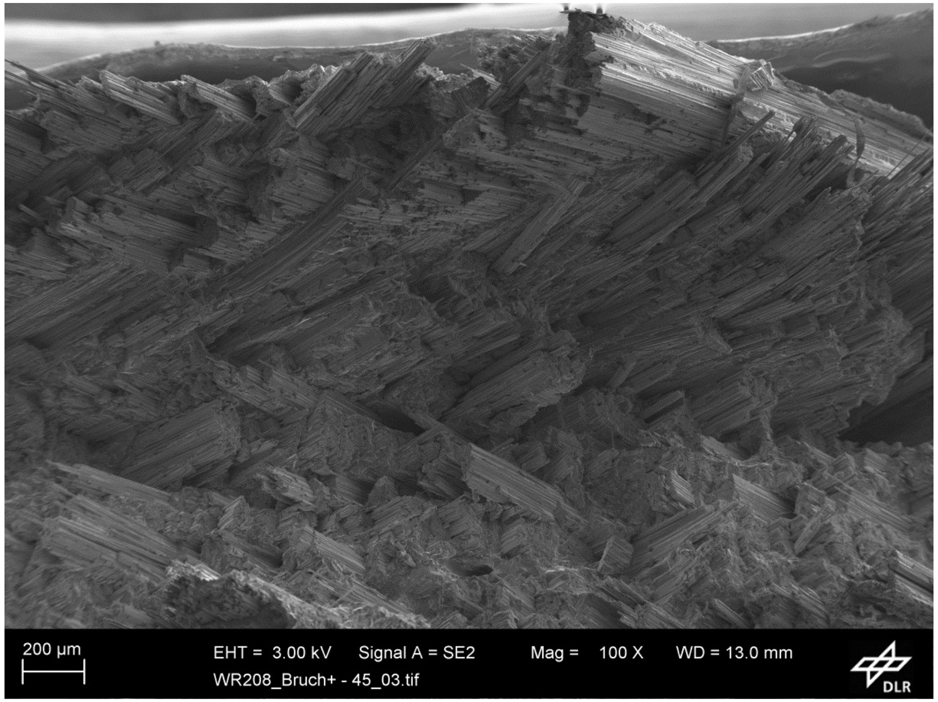
Fracture Surfaces with Fibre Orientation to Loading Direction (±30°)

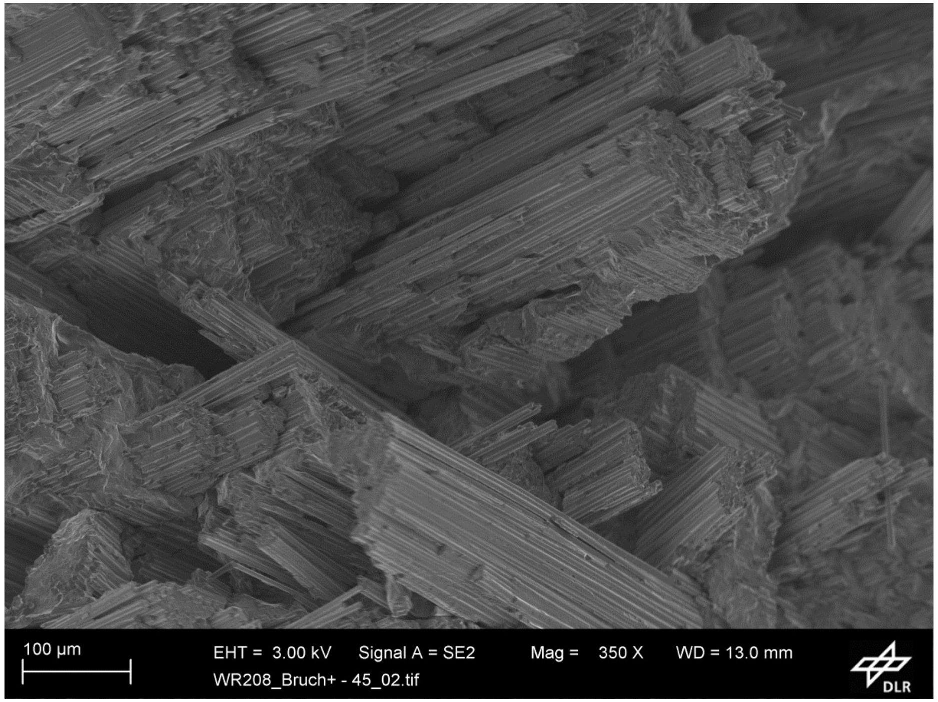




→ rather low fibre pull-out

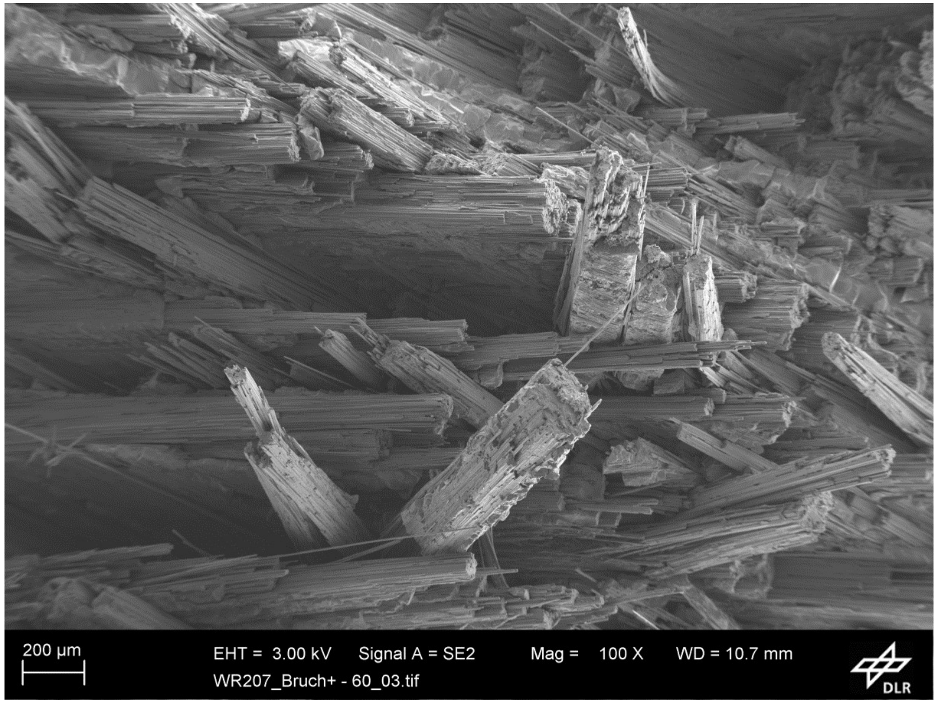
Fracture Surface with Fibre Orientation to Loading Direction (±45°)

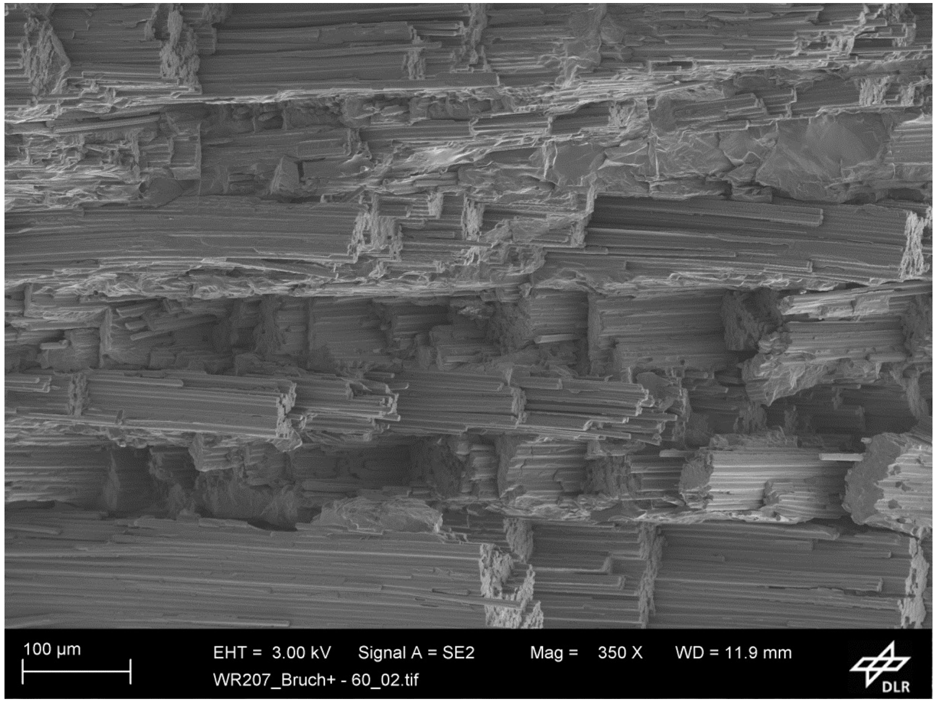




→ fibre pull-out and matrix disintegration

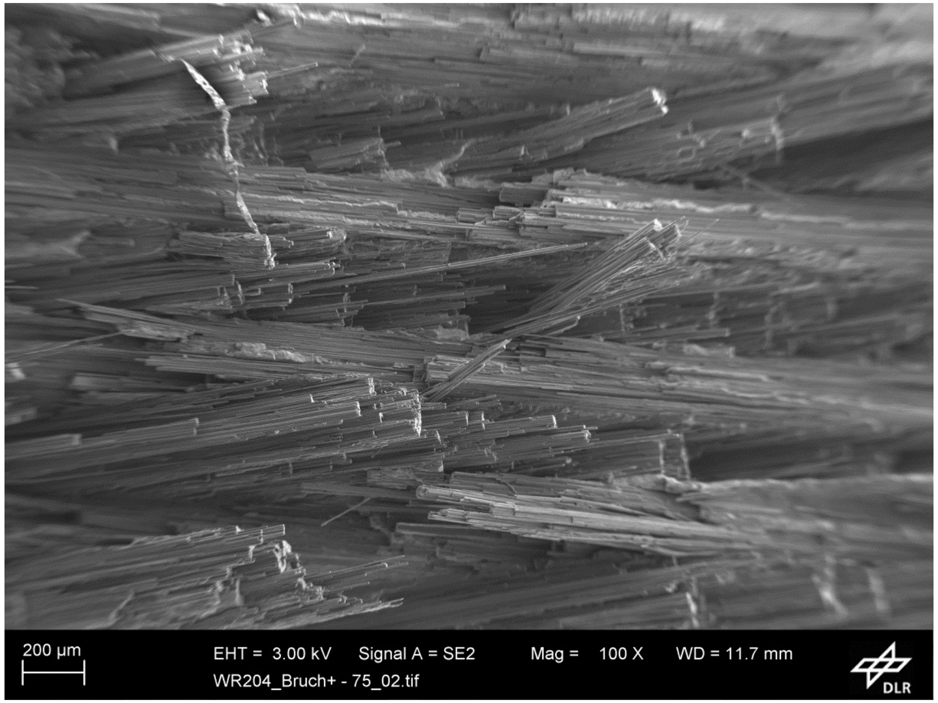
Fracture Surface with Fibre Orientation to Loading Direction (±60°)

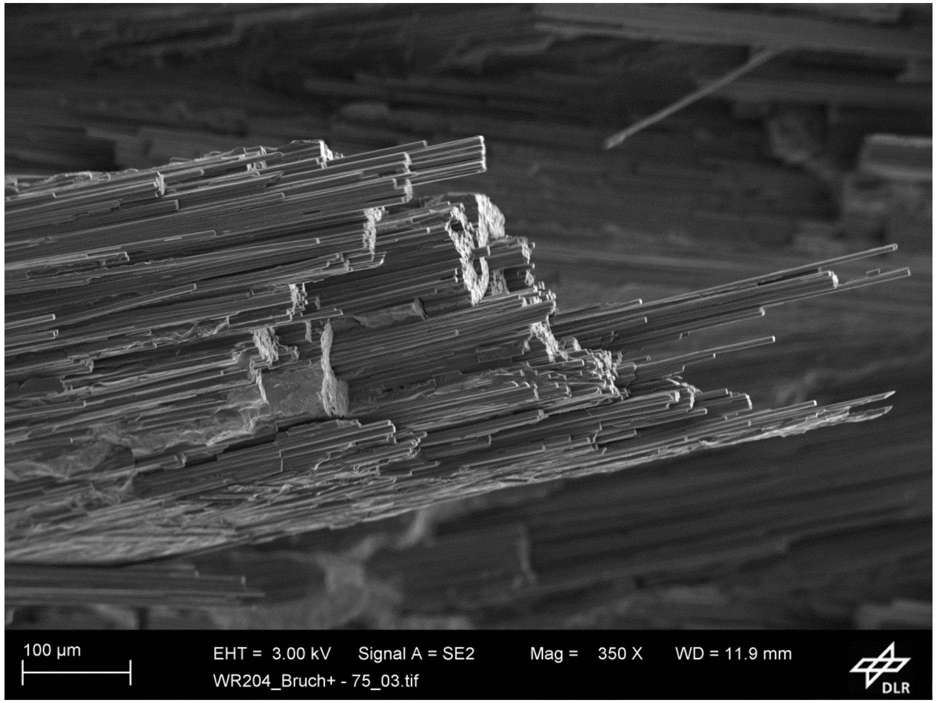




→ fibre pull-out and rather high matrix disintegration

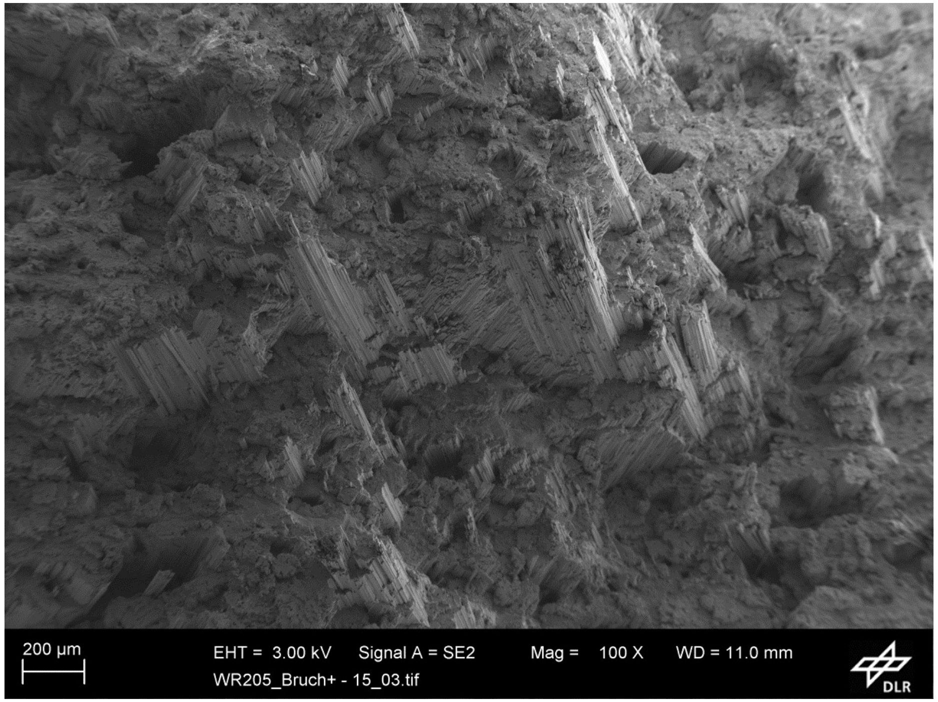
Fracture Surface with Fibre Orientation to Loading Direction (±75°)

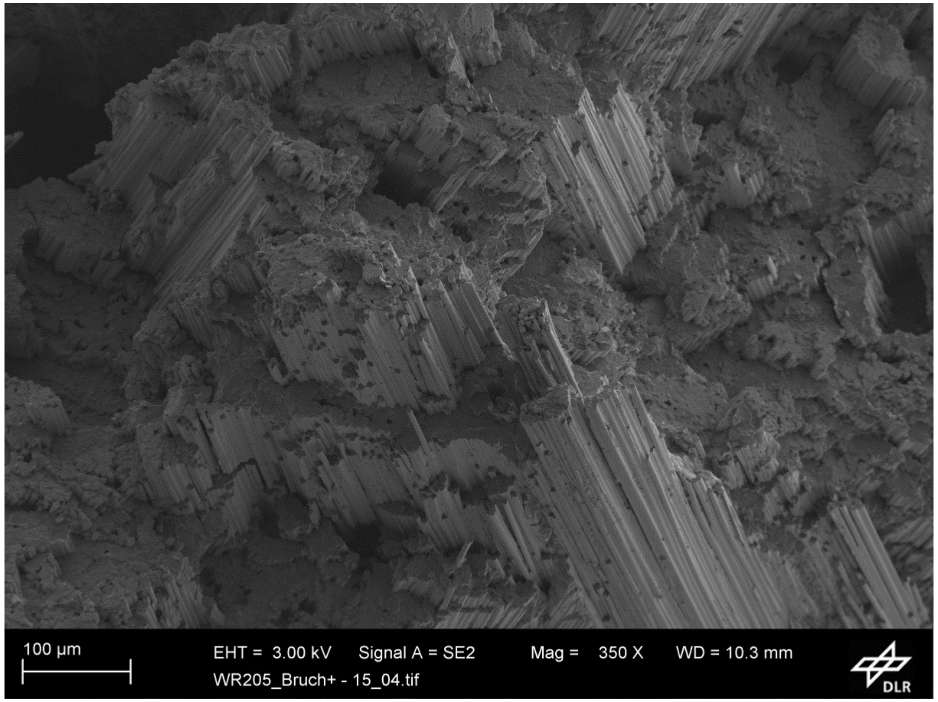




→ high amount of matrix disintegration, almost no matrix visible

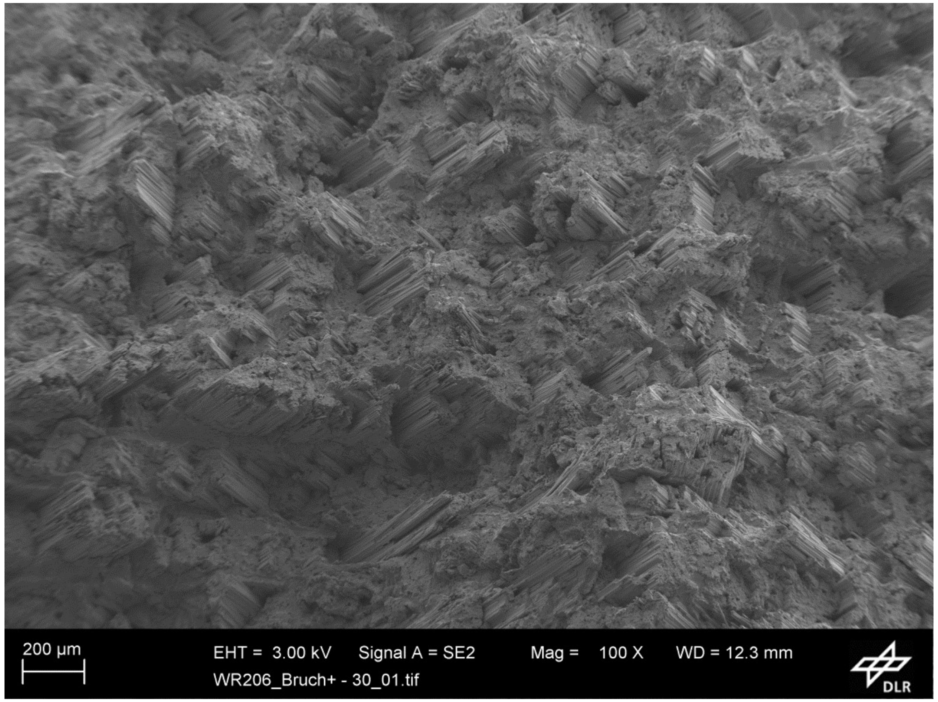
Fracture Surface after Tensile Testing at 1300°C (±15°)

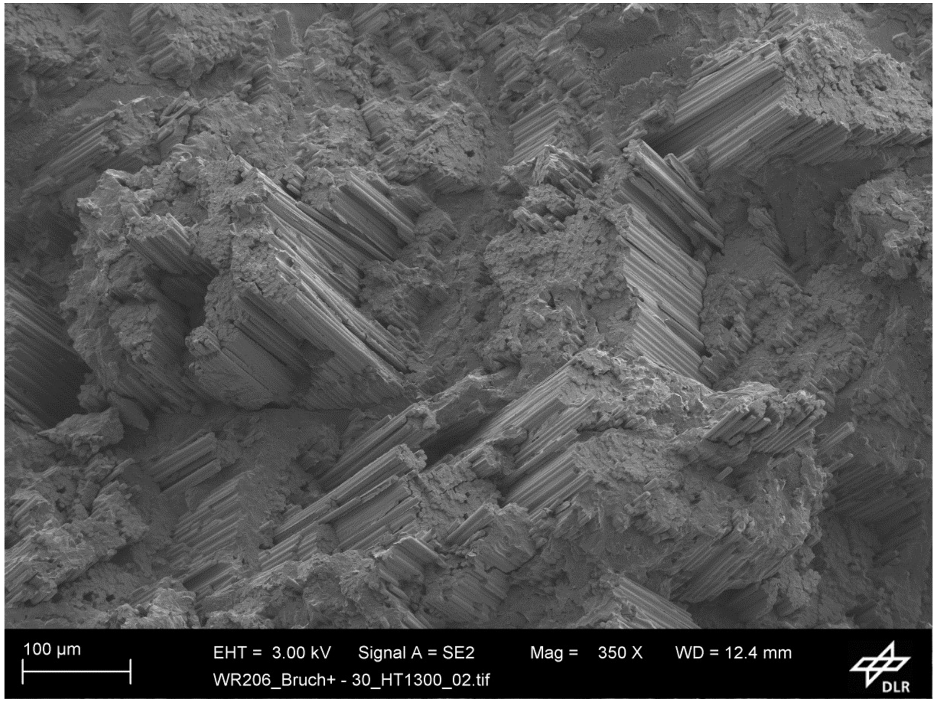




→ rather high fibre pull-out

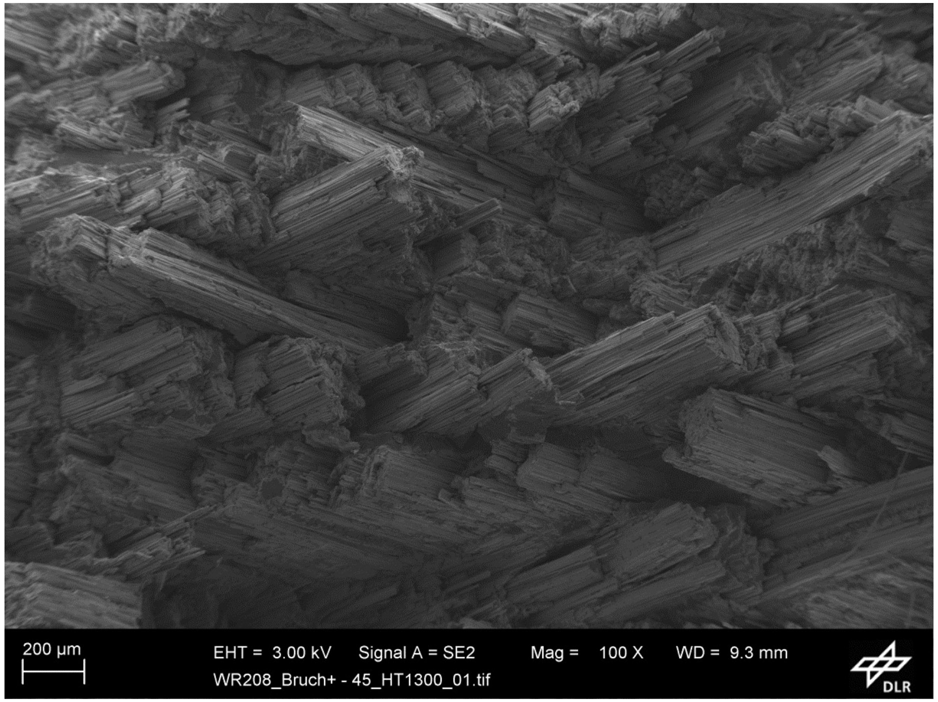
Fracture Surface after Tensile Testing at 1300°C (±30°)

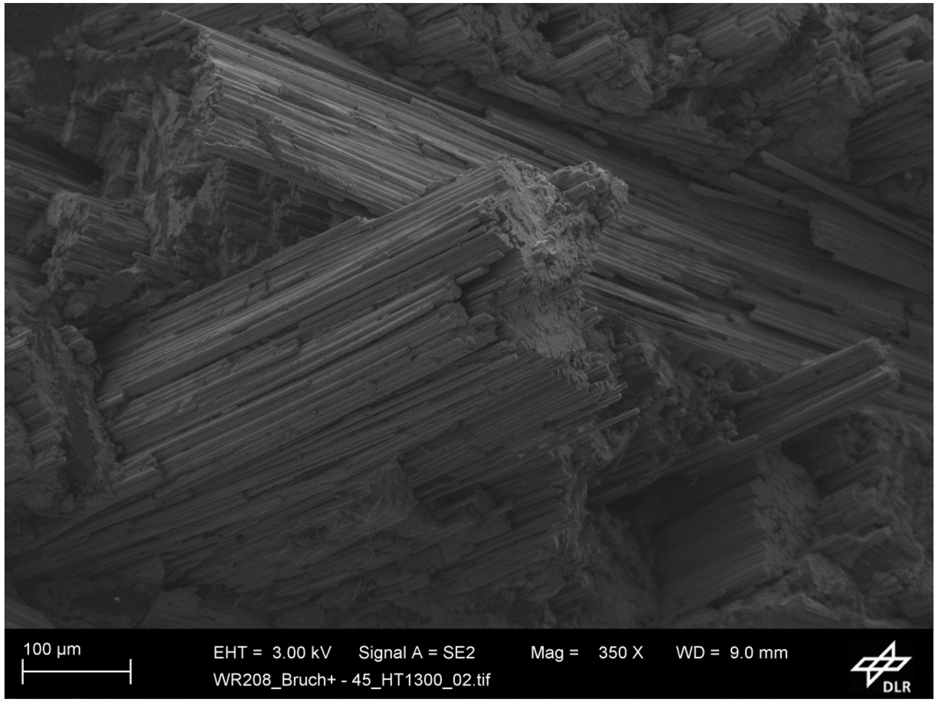




→ rather low fibre pull-out

Fracture Surface after Tensile Testing at 1300°C (±45°)





→ fibre pull-out and matrix disintegration, almost no matrix visible

Conclusions

- Water-based phenolic resins (MF43) and C-fibres (T800) were successfully applied in wet filament winding and LSI-processing

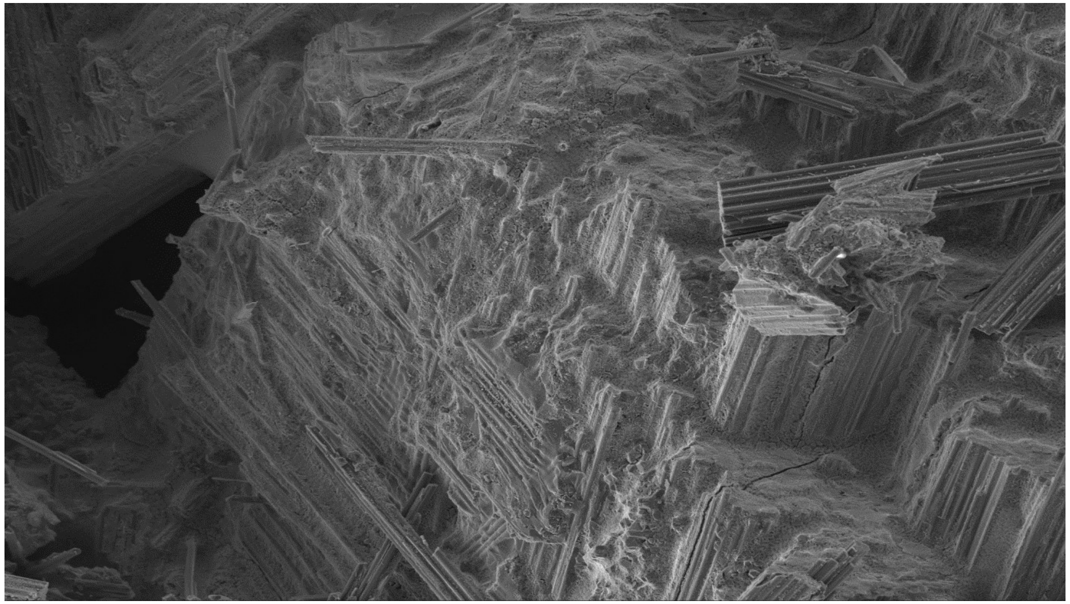
- Almost dense C/C-SiC composites could be obtained via autoclave processing without applying any mechanical forces in a four step process

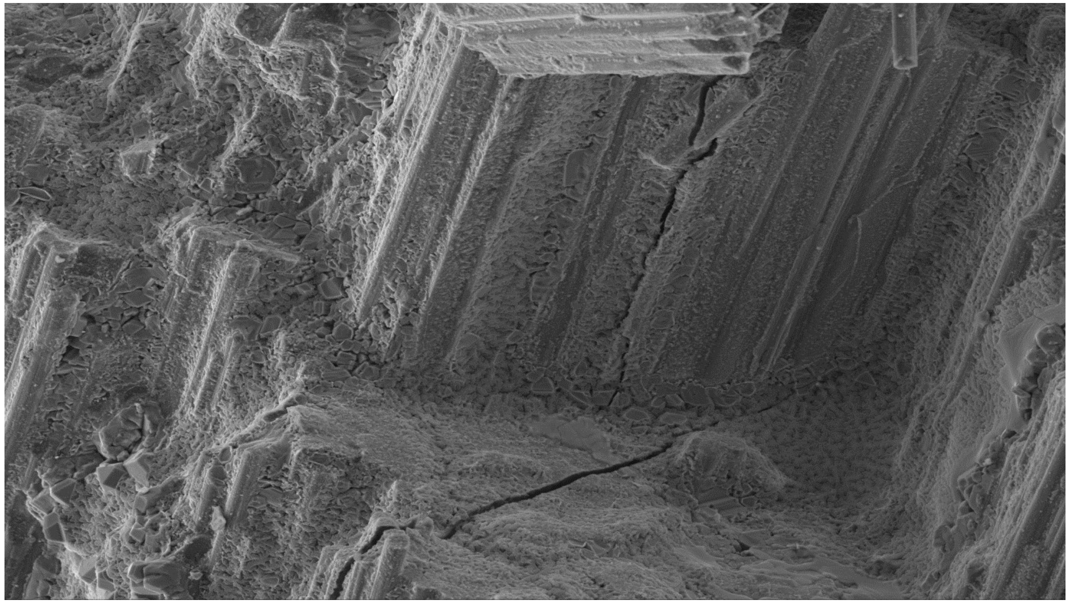
- Thermomechanical properties strongly depend on fibre orientation as expected, thus calculation tools for composites such as classical laminate or inverse laminate theory can be applied for structural evaluation and prediction of mechanical performance of C/C-SiC structures

- First investigations of fracture surfaces can interprete the fracture behaviour in correlation with composite strength: high fibre pull-out leads to high strength and stiffness (±15°); high matrix disintegration to low strength and stiffness (±75°); fracture strain as superimposition of both above is more complex

- No significant decrease in mechanical properties at high temperatures (1300°C) could be measured so far, which makes C/C-SiC interesting as a low cost, damage-tolerant high temperature material for high temperature applications, such as rocket motors, combustion chambers, etc. with graceful failure

Outlook:
Fracture Surfaces after Tensile Testing at 1600°C on a First Test Sample (tensile strength = 92 MPa, no measure of strain possible)





→ Further mechanical testing at high temperatures and characterisation will be performed pretty soon!
→ Where is the limit? Is the limit determined by strength or stiffness?

Acknowledgement

- The authors are pleased for greatful financial support by the German Ministry of Defense as well as fruitful discussions with many colleagues DLR internal as well as colleagues of Technical University of Aachen, and last but not least …

- Many other technical persons from specialized companies who made this unique testing facility Indutherm become working successfully.

- And you for your kind attention!

- Any questions?