C/C-SiC Materials Based on High Performance C Fibres with Tailored Fibre-Matrix Bonding

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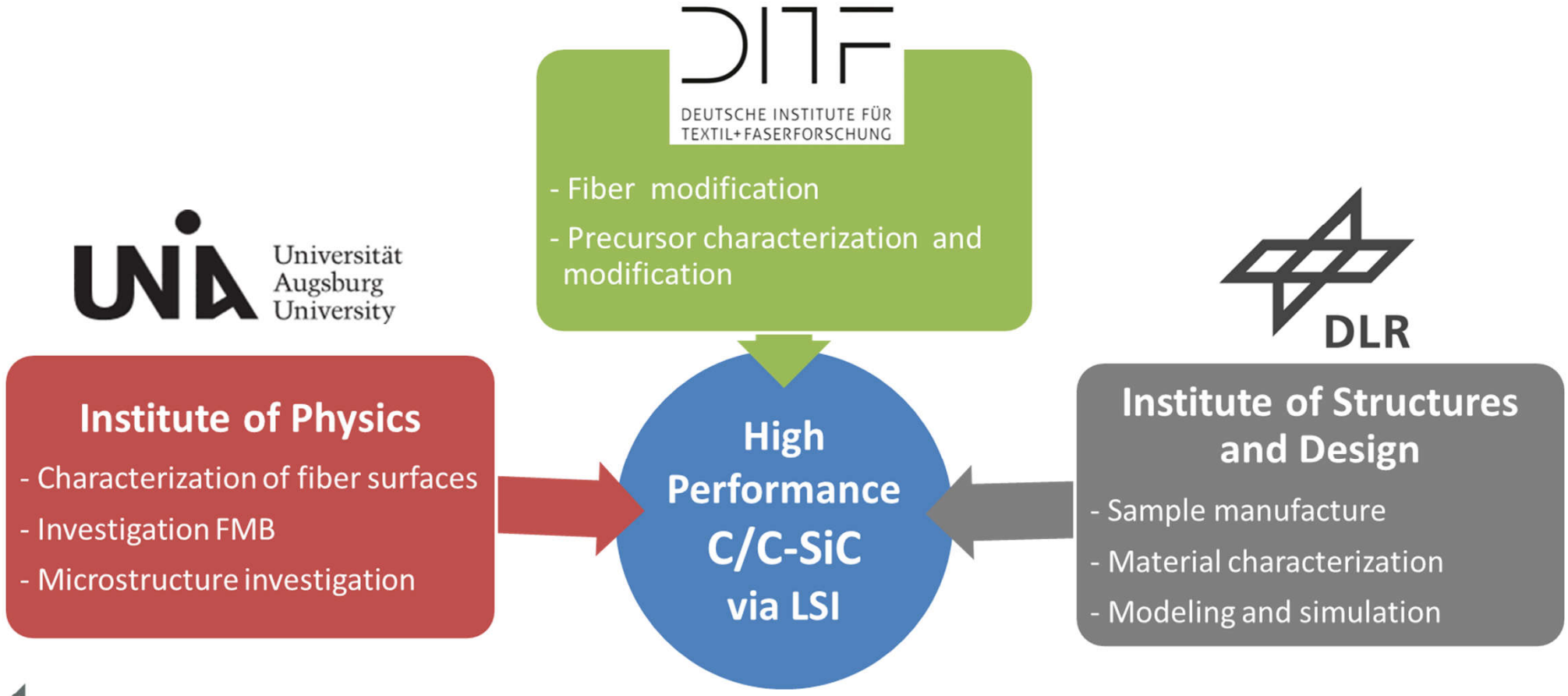
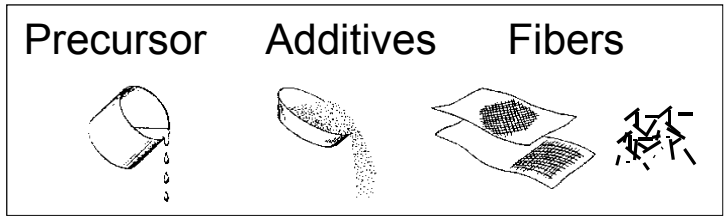
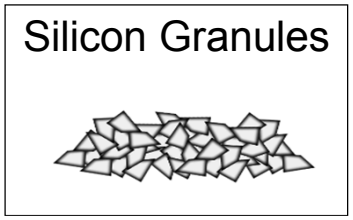
DFG-Project KeraFaM - High Modulus and High Strength C/C-SiC Ceramic Matrix Composites by Fiber-Matrix Interface Tailoring via Modification of Fiber Surface and Matrix Precursor



Liquid Silicon Infiltration Process LSI





CFRP (Shaping)

T_{max} = 195 °C / 240 °C

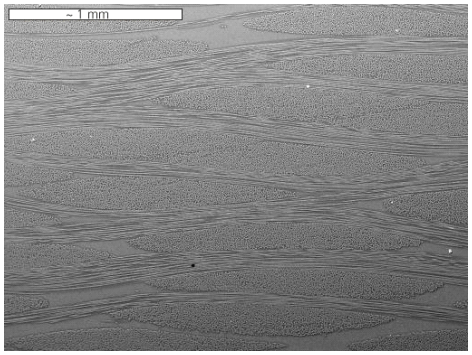
Pyrolysis

T_{max} = 1 650 °C, N_2

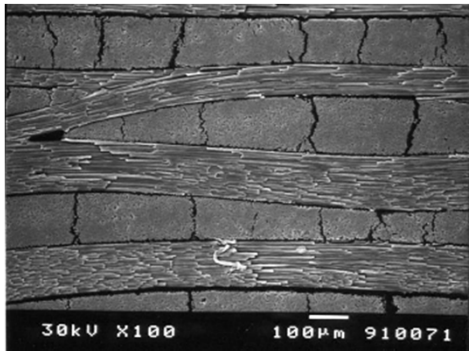
Siliconization

Si + C → SiC

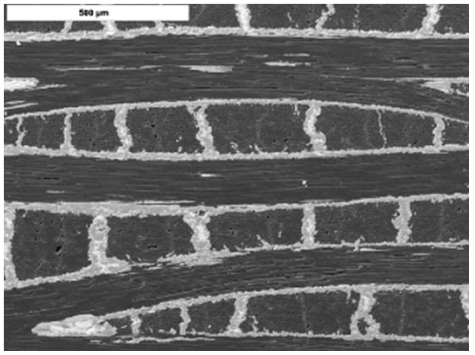
T_{max} = 1 650 °C, vacuum



CFRP



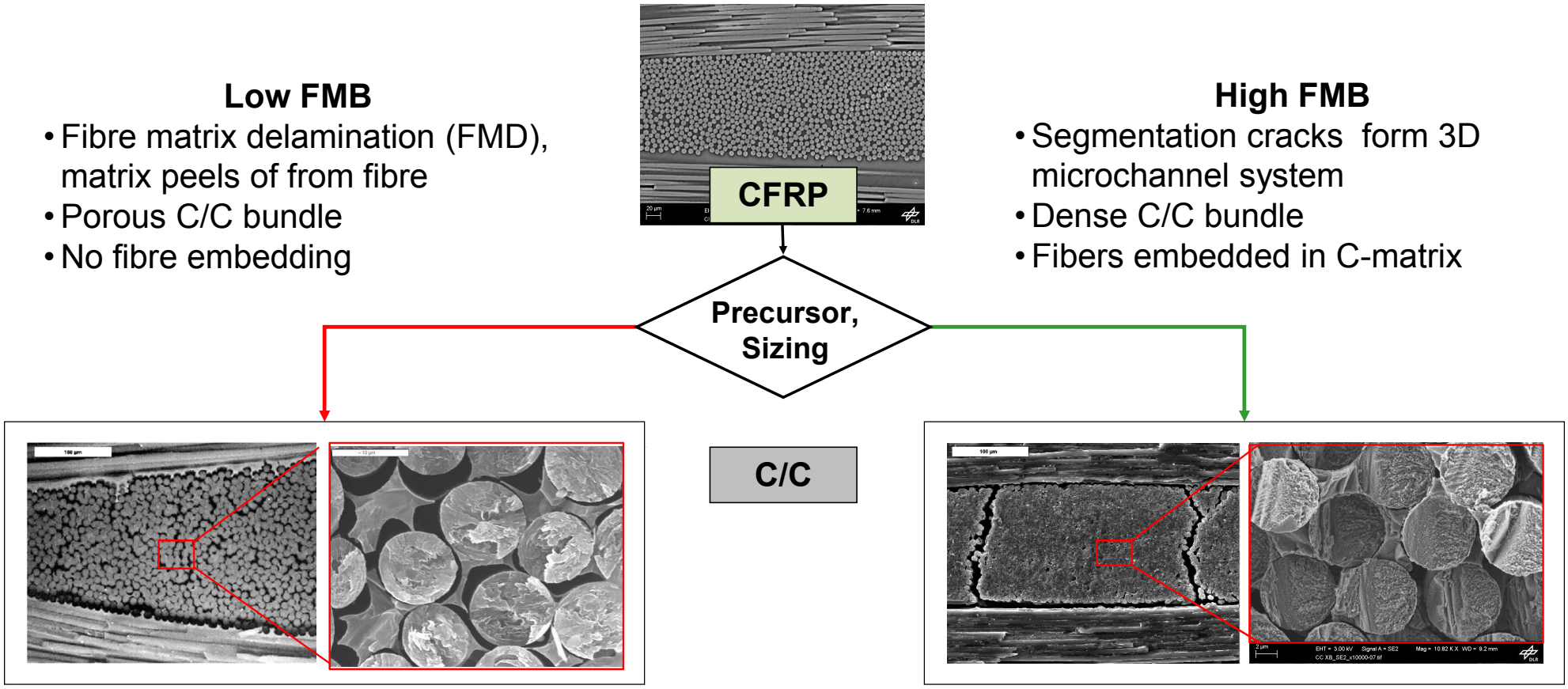
C/C



C/C-SiC

- No fibre coating
- Microstructure and material properties depend on fibre matrix bonding (FMB)

C/C-SiC Materials Based on Low and High Fiber Matrix Bonding (FMB)



Low FMB

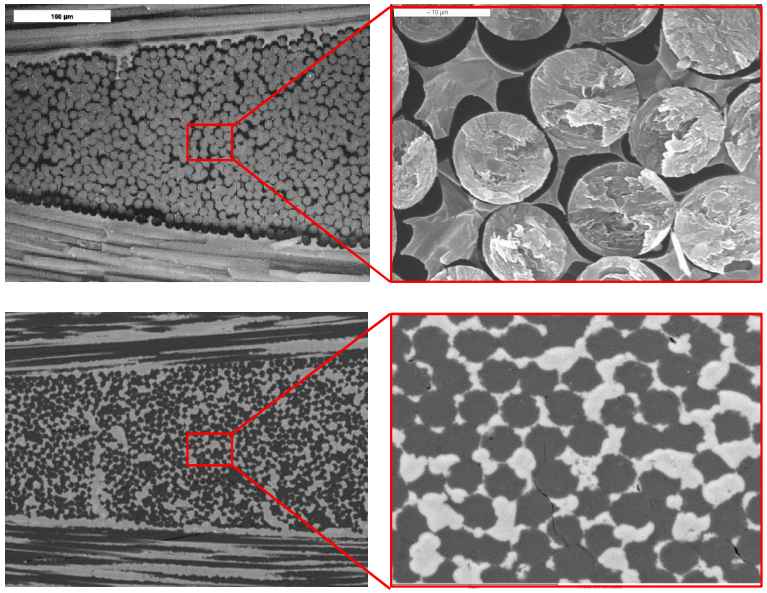
- Fibre matrix delamination (FMD), matrix peels of from fibre
- Porous C/C bundle
- No fibre embedding

High FMB

- Segmentation cracks form 3D microchannel system
- Dense C/C bundle
- Fibers embedded in C-matrix

C/C-SiC Materials Based on Low and High Fiber Matrix Bonding (FMB)

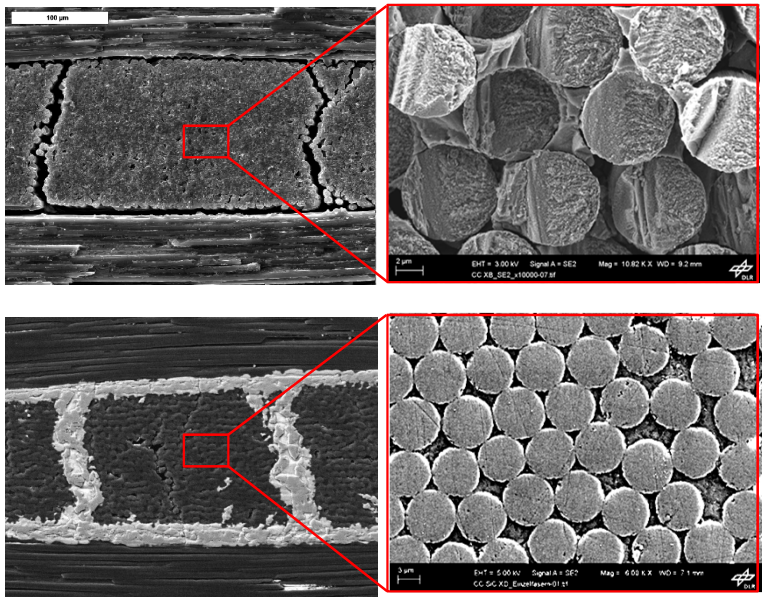
Low FMB



C/C

C/C-SiC

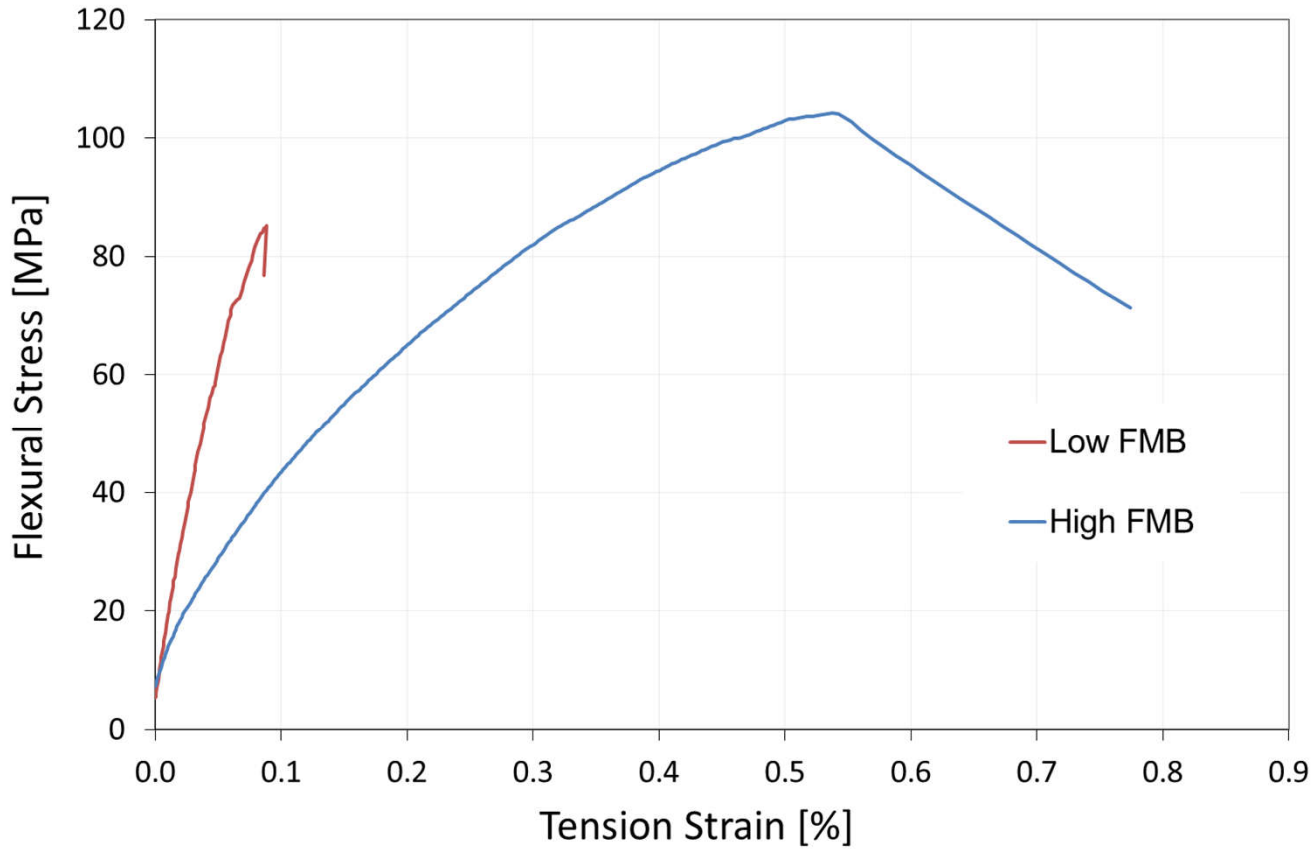
High FMB

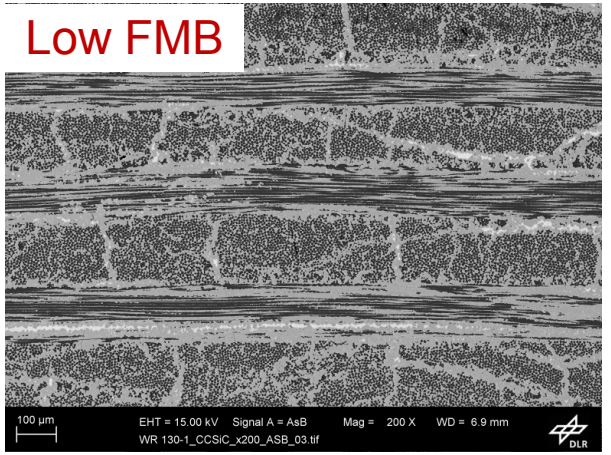


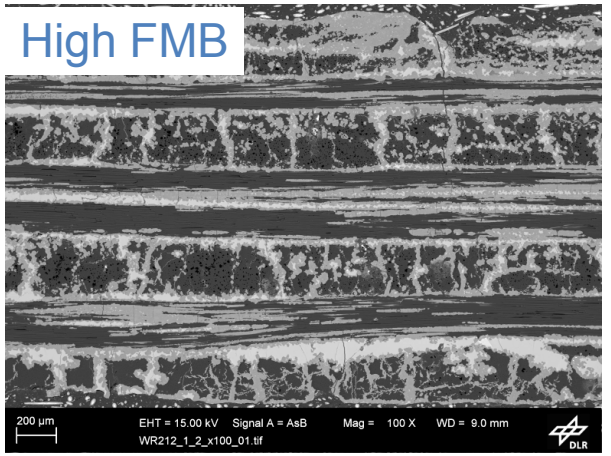
- Si infiltrates whole fiber bundle
- High conversion rate of fibres / SiC content
- High brittleness / low fracture strain

- Si infiltrates microchannel system, only
- Low conversion rate / limited fiber damage
- Increased fracture strain / damage tolerance

C/C-SiC Materials Based on Low and High Fiber Matrix Bonding (FMB)

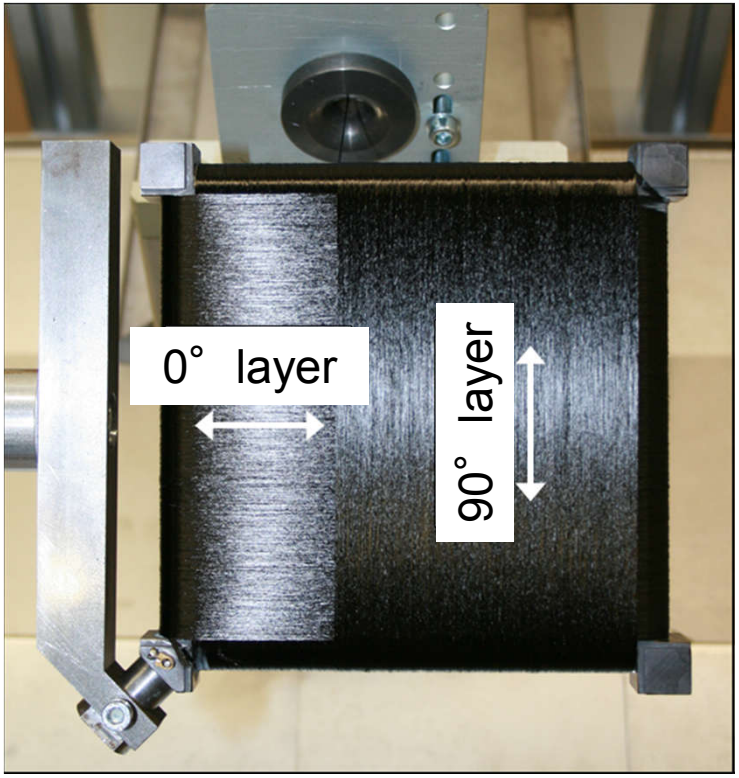






CFRP Preform Manufacture

- Wet filament winding of C fibre roving
- 0° / 90 ° cross-ply laminates (120 x 120 mm²)
- Densifying of laminate (d = 2.2 mm)
- Curing of precursor (15 bar, 195 °C)
- Cutting of plates and demoulding from core mandrel
- Thermal treatment (tempering) in furnace (1bar , 240 °C)
- Pyrolysis (1650 °C, N_2)
- Siliconization (1650 °C, vacuum)



Precursors

		JK 60	MF 43
Resin type		Phenol-Resol	Phenol-Resol
Solvent		Acetone	Water
Solvent Content	[mass.-%]	< 25	3
Nonvolatile portion (135 °C)	[mass.-%]	70 ± 2	79 ± 2
Viscosity (20 °C)	[mPas]	1000 ± 200	2600 ± 200
Phenolic content	[mass.-%]	1.9	20 - 25
C yield (900 °C)	[mass.-%]	42	47
Selection criteria		Standard resin for high FMB (autoclave, pressing)	• High C yield, low mass loss during curing • No critical outgassing during curing

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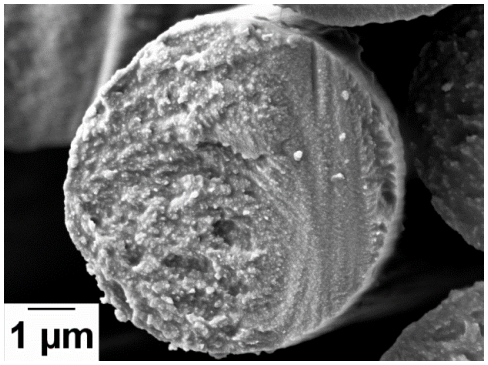
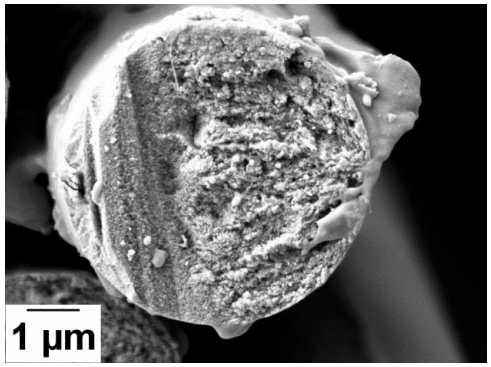
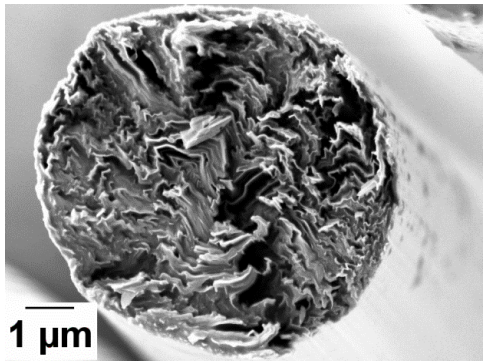
Fibres

Fibre type		HT		UHT		UHM	
Fibre		HTA HTA 40 E13, 6K		T1000 T 1000 GB, 12K		YS 90 A Granoc YS-90A-30S, 3K	
Manufacturer		Tejin Tenax		Toray		Nippon Graphite Fibres (NGF)	
Fibre precursor		PAN		PAN		Pitch	
Tensile Strength	[GPa]	3.03	4.4	5.1	6.37	-	3.53
Young's Modulus	[GPa]	172	240	272	294		880
Ultimate Strain	[%]	1.7	1.7	1.8	2.2	-	0.3
Filament Diameter	[µm]		7		5		7

manufacturers` data



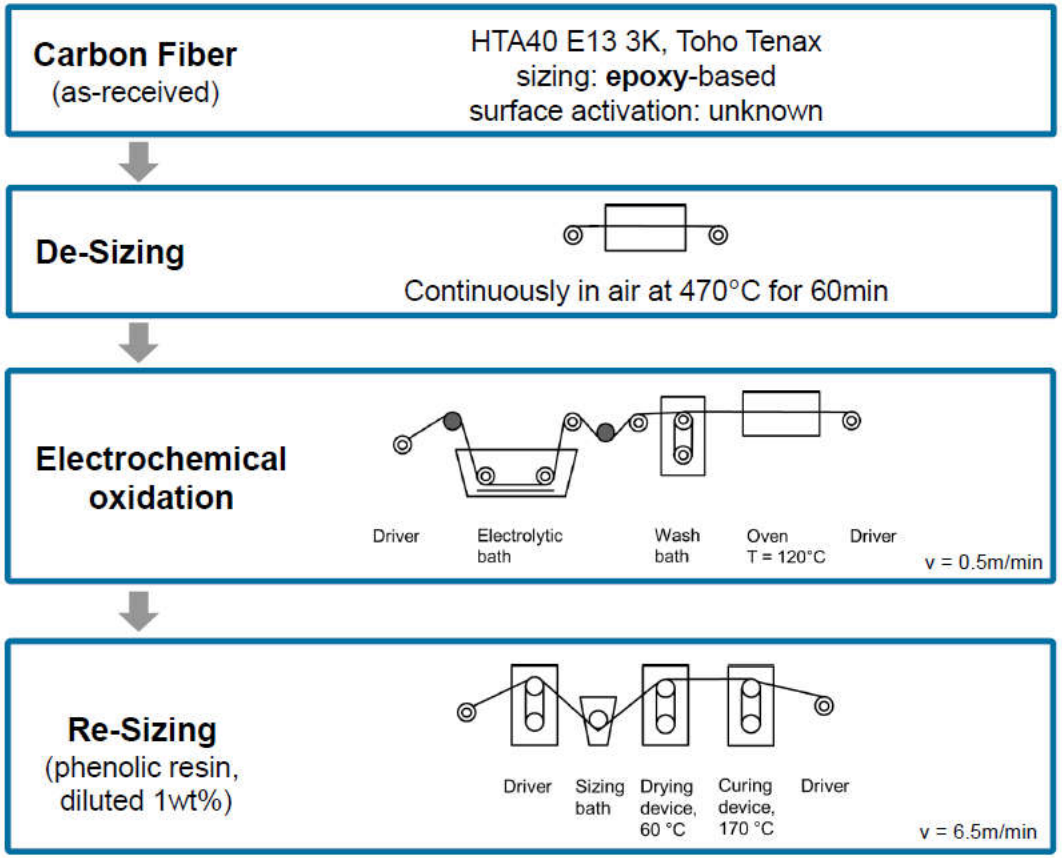




DLR

Fibre Variants

HTA
HTA - DES - SIZ
HTA - DES - EO - SIZ
T1000
T1000 - DES - SIZ
T1000 - DES - EO - SIZ
YS90



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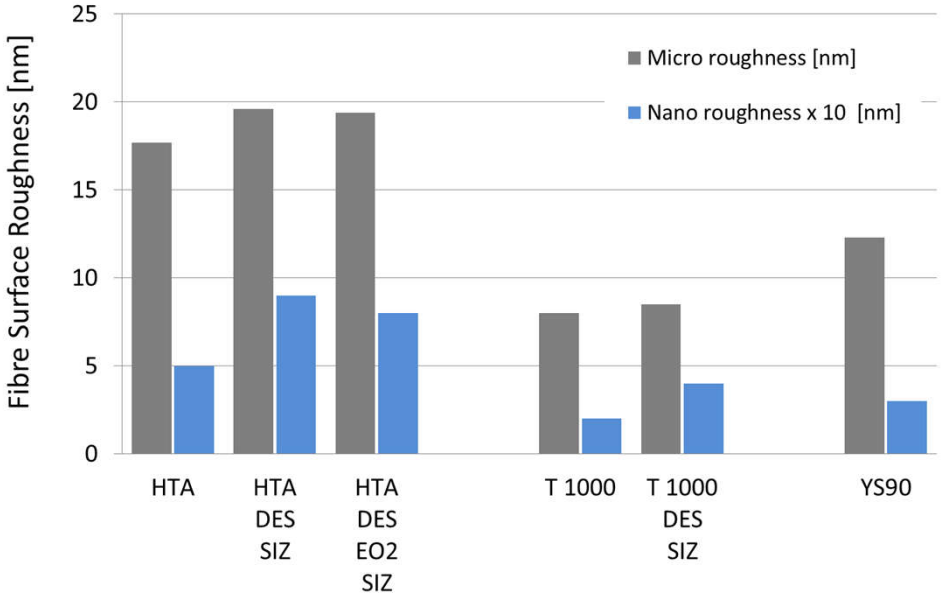
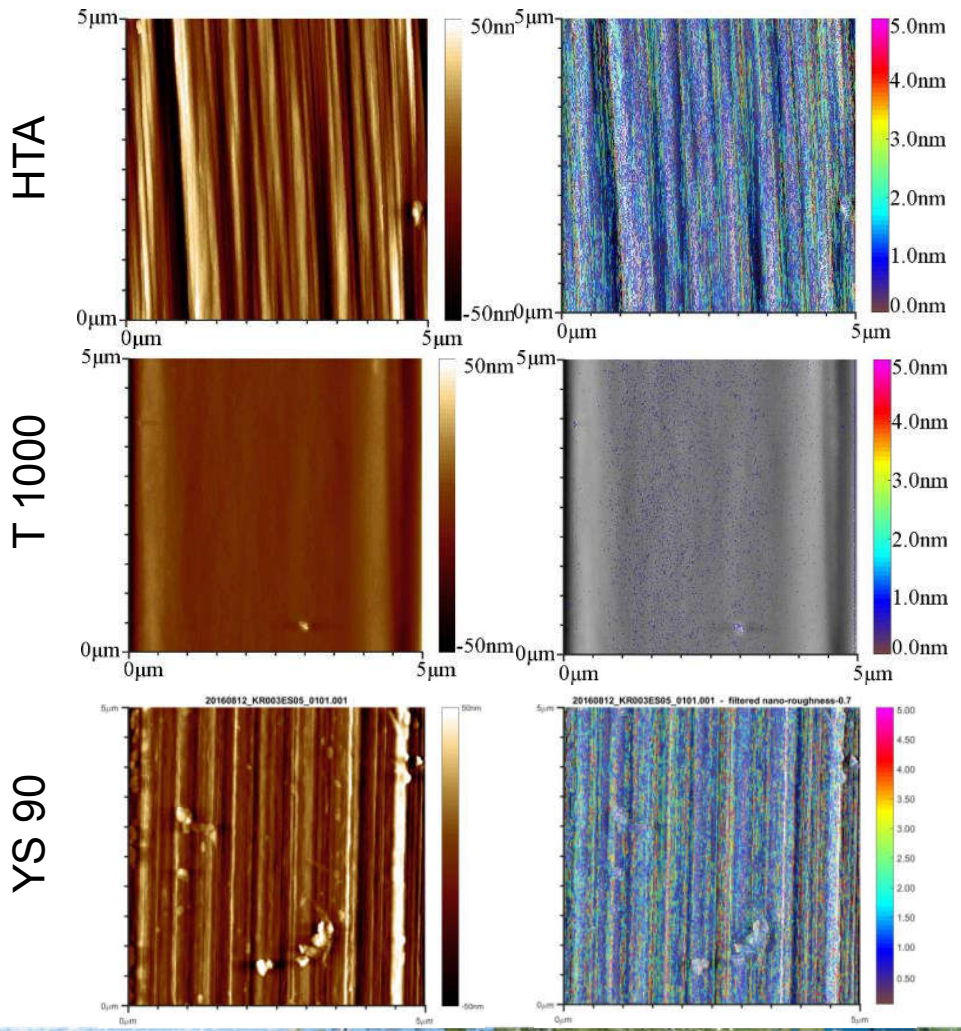
Fibre Surface Roughnesss (AFM)





DLR

Sample Geometry

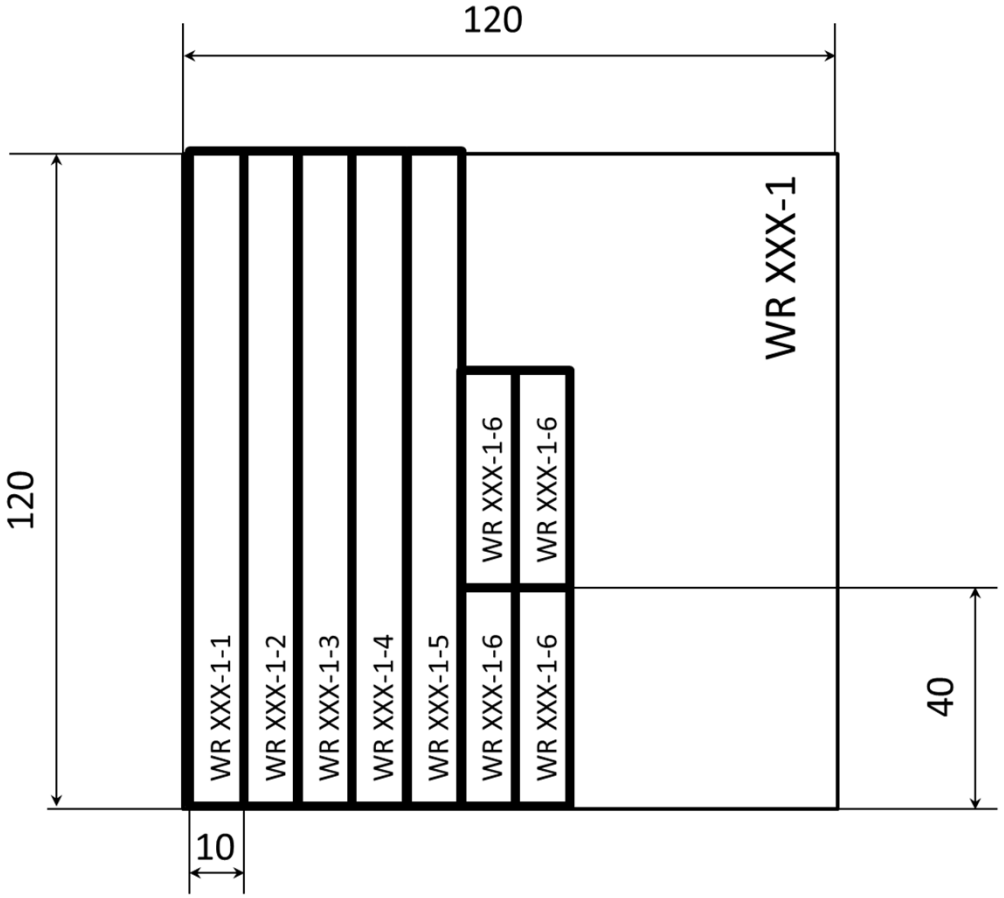
Tension (DIN 658-1)

5 samples a` 120 x 10 x 2 mm³

Three point bending (DIN 658-3):

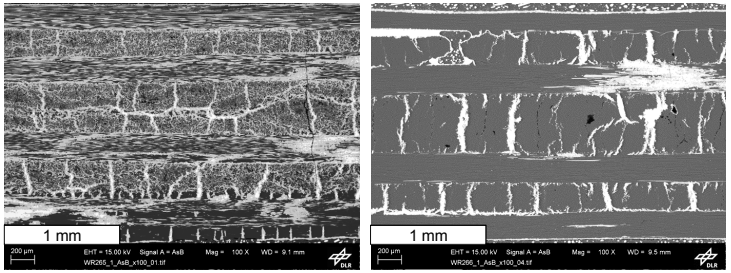
4 samples a` 40 x 10 x 2 mm³

Microstructure analysis (SEM, ImageJ)

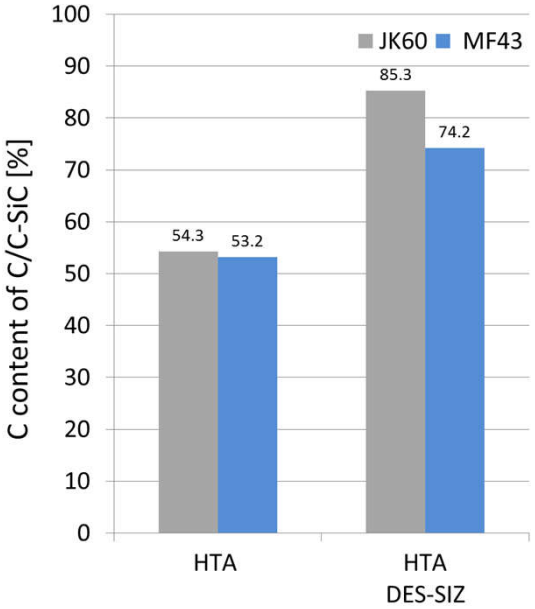


C/C-SiC Microstructures – HTA

JK 60

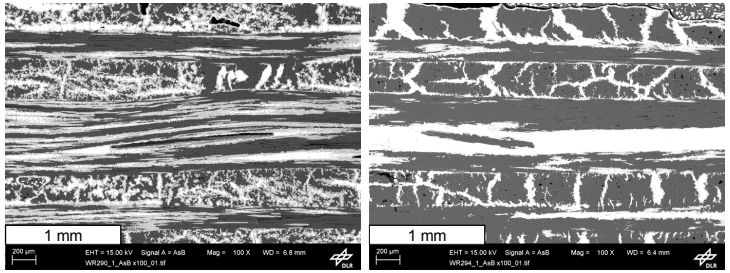


HTA HTA-DES-SIZ



Grey scale analysis (ImageJ)

MF43



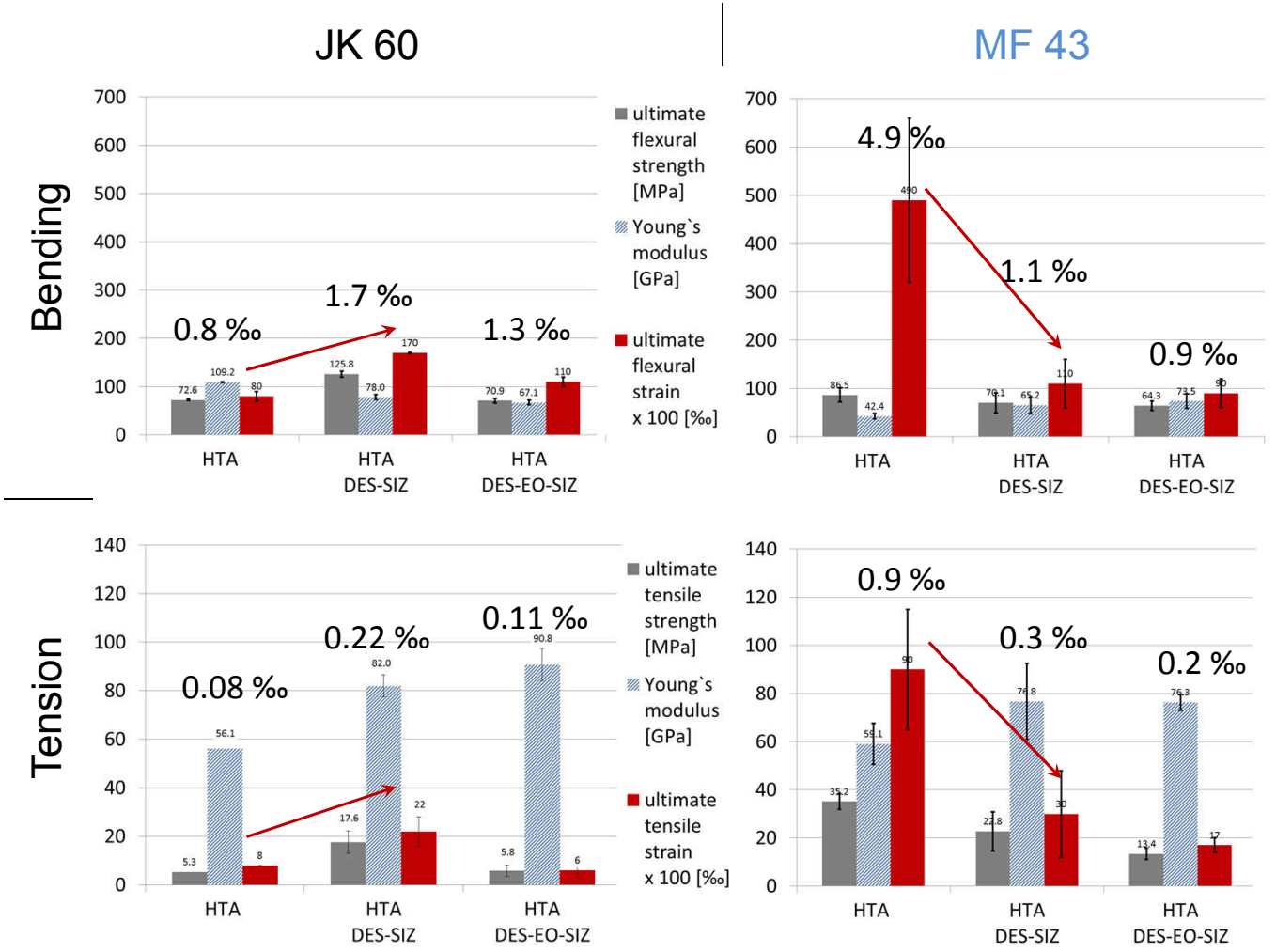
HTA HTA-DES-SIZ

Resizing of HTA fibers with matrix precursors leads to:

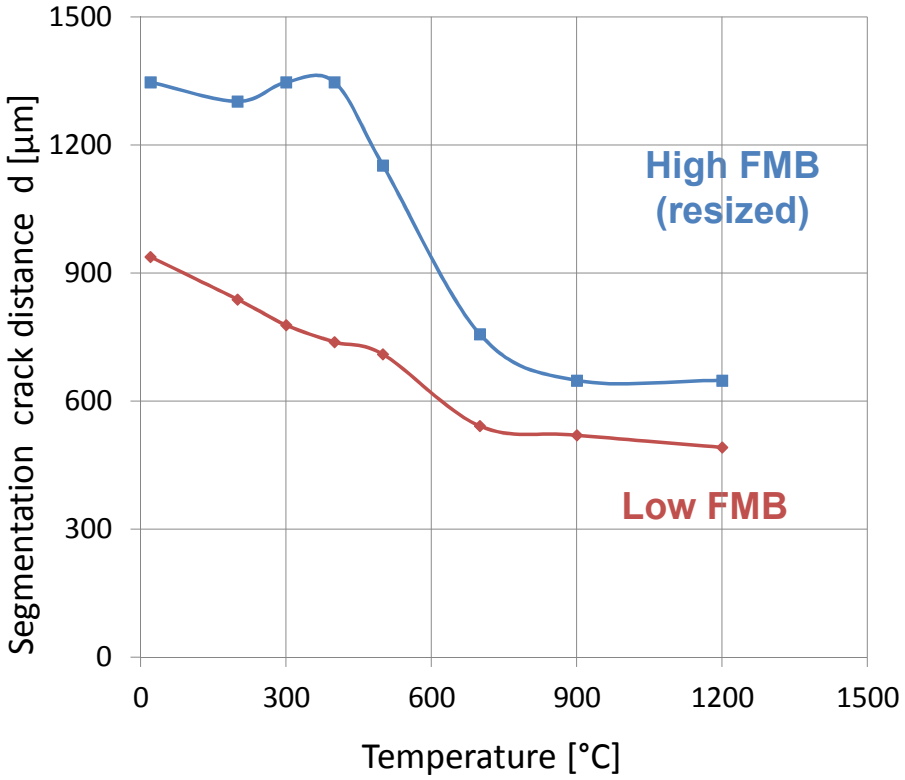
- Dense C/C bundles
- Decreased conversion rate / increased C-content

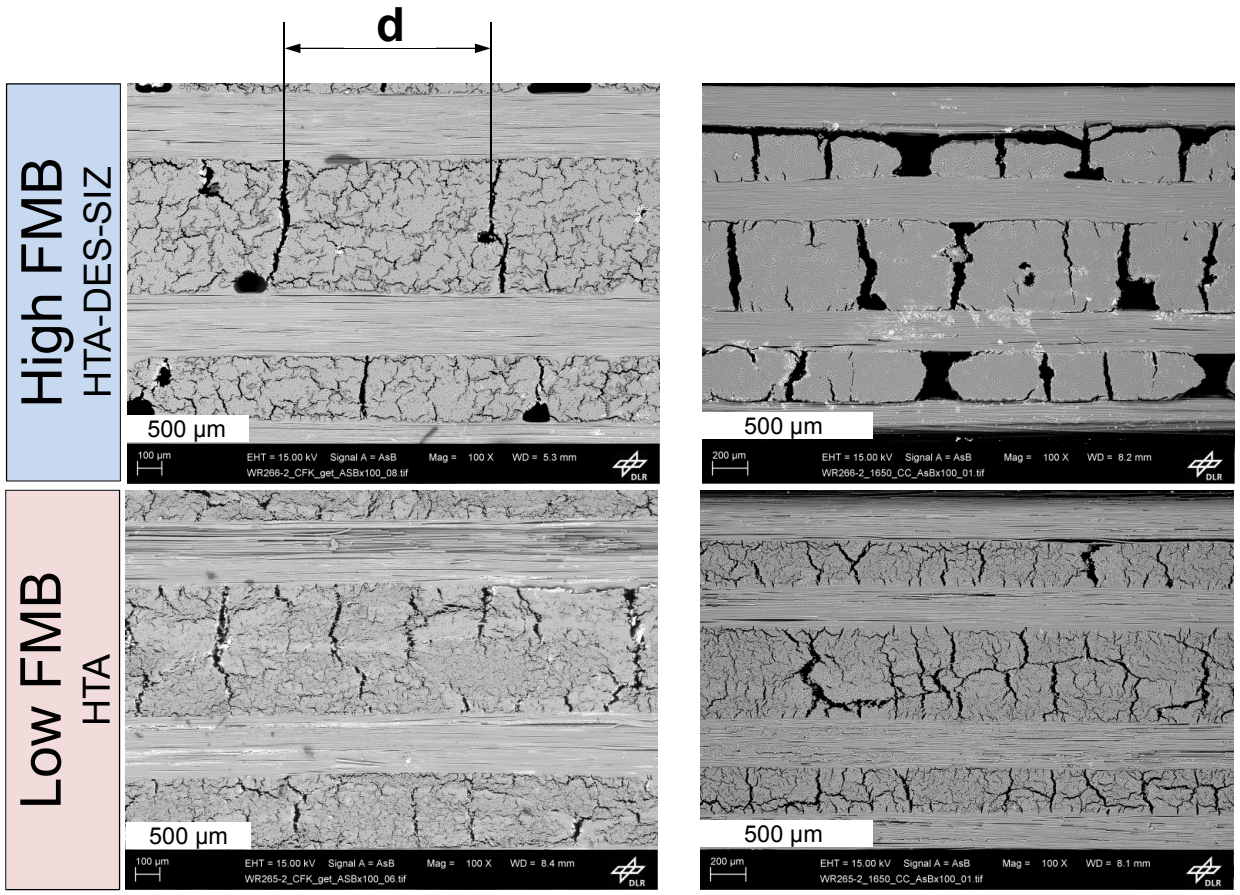
Results – HTA

- Highest strain (4.9 / 0.9 ‰) with MF43 Precursor and unmodified fibre
- Resizing with JK 60 → increased properties (ε_t: 0.08 → 0.22 ‰)
- Resizing with MF43 → decreased strain and strength (ε_t: 0.9 → 0.3 ‰)
- Minor influence on modulus
- Fibre oxidation compensates resizing effect or decreases properties



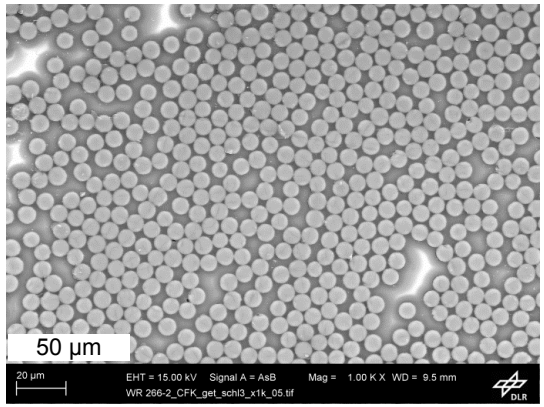
Development of Segmentation Cracks (JK60)

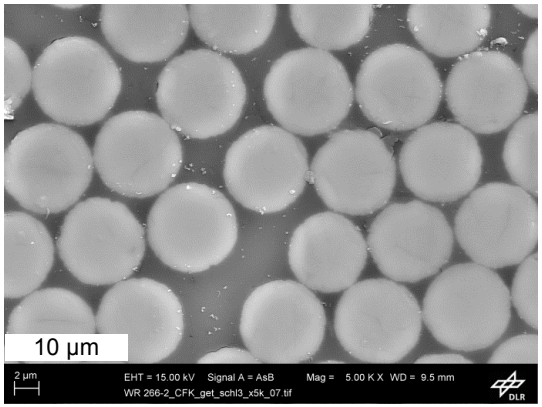


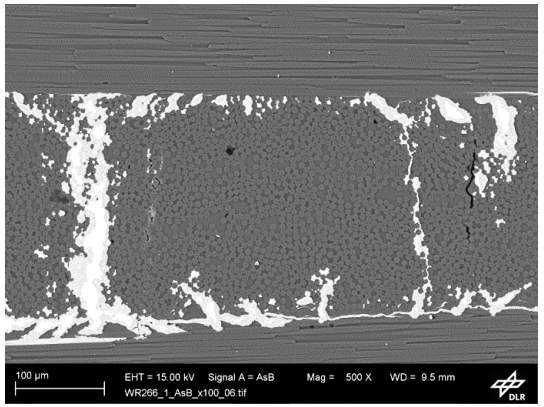


Fibre Matrix Delamination (FMD) in CFRP (JK60)

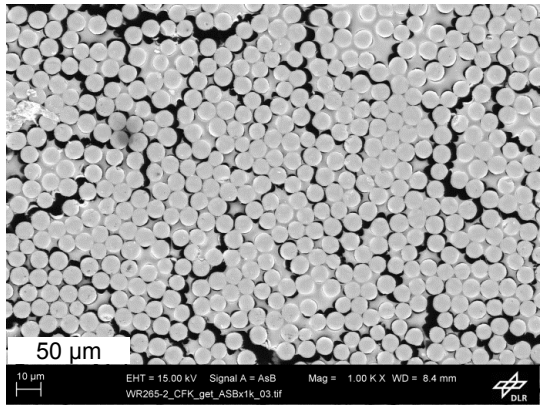
High FMB
HTA-DES-SIZ

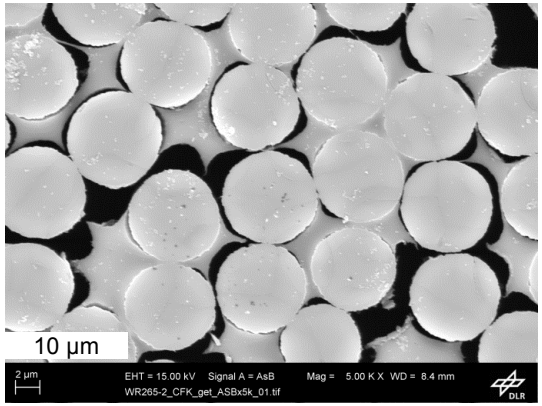


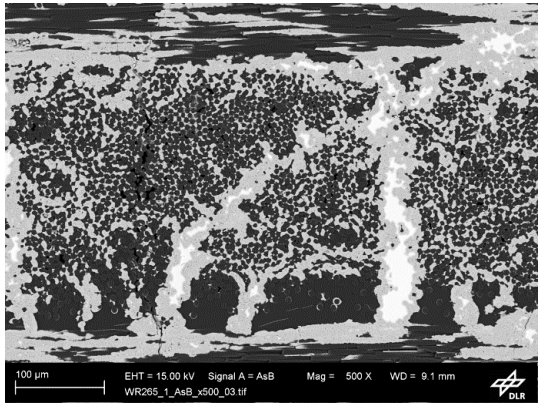




Low FMB
HTA





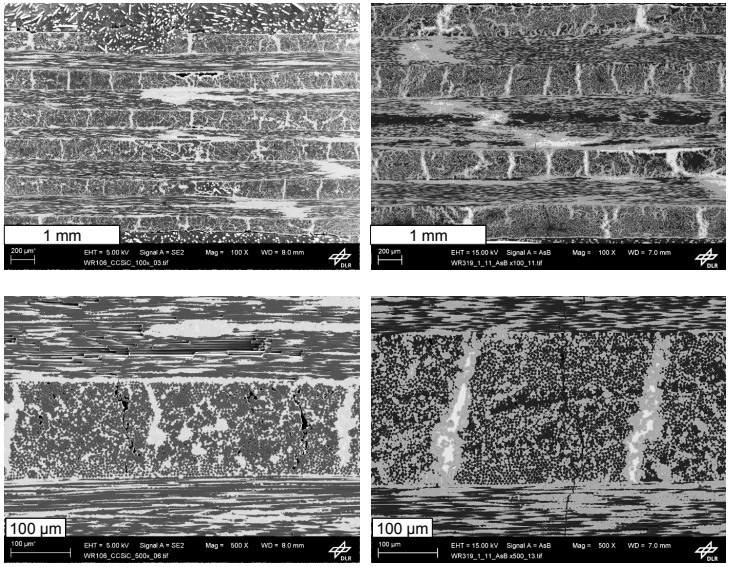


CFRP (240 °C)

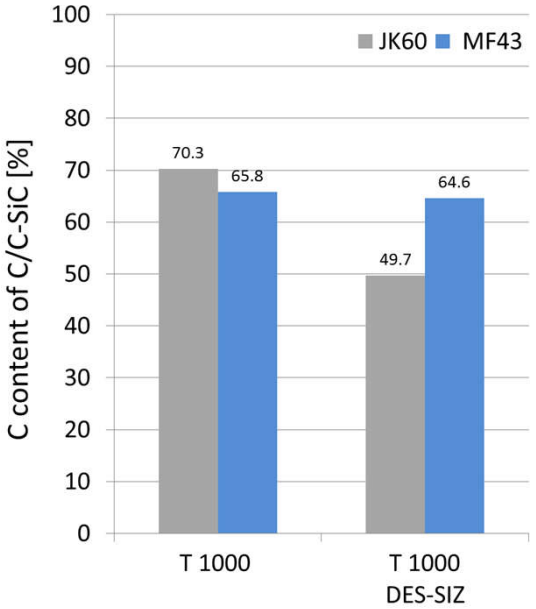
C/C-SiC

C/C-SiC Microstructures – T 1000

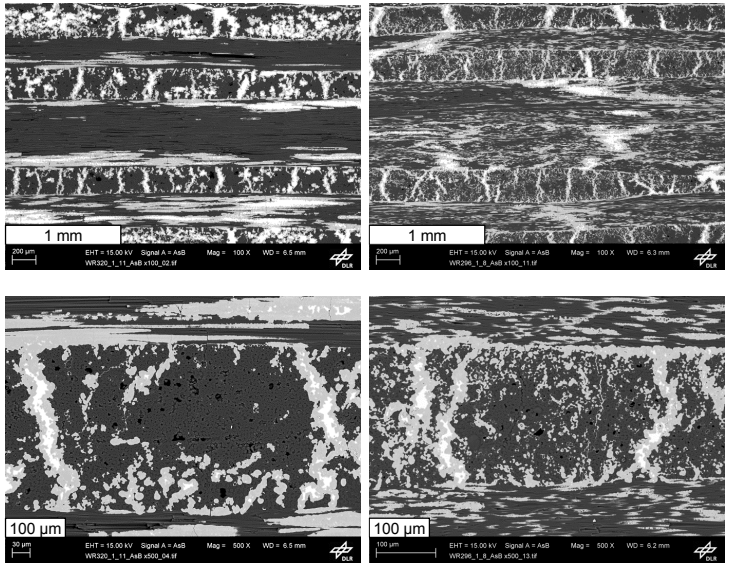
JK 60



T 1000 T 1000-DES-SIZ



MF 43



T 1000 T 1000-DES-SIZ

Resizing of fibres with precursors leads to:

- Decreased C-content and increased conversion rate (JK60)
- Similar C-content but higher conversion rate due to homogeneous SiC distribution (MF43)

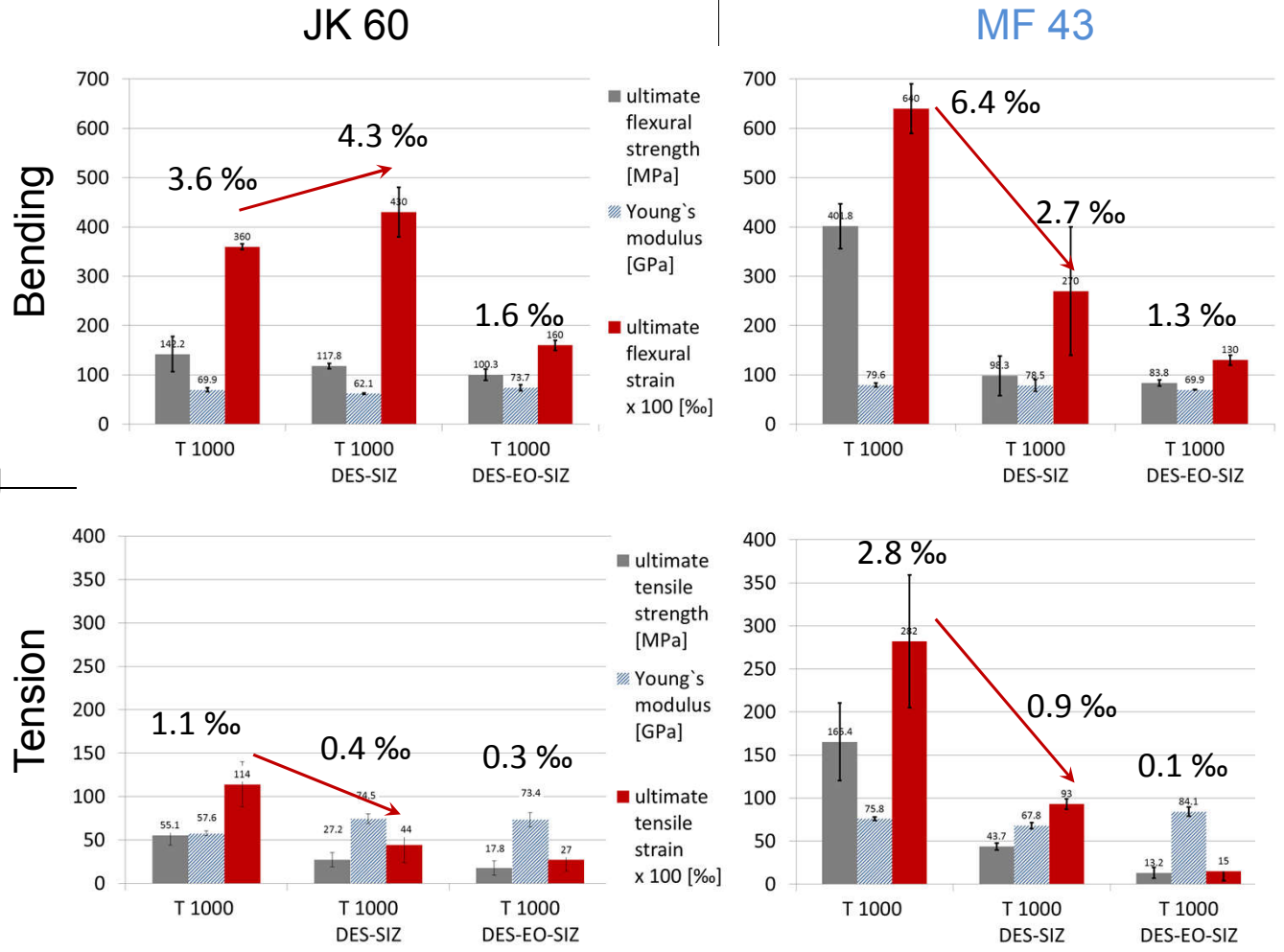
Results – T 1000

- Higher strain and strength compared to HTA

  ε_t = 2.8 ‰; σ_t = 165 MPa; E_t= 76 GPa

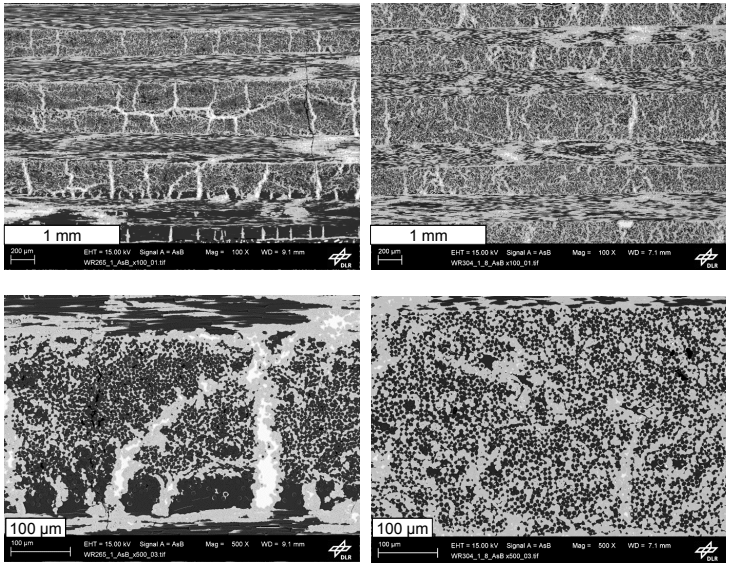
  ε_b= 6.4 ‰; σ_b= 401 MPa; E_b= 80 GPa

- JK 60: Inconsistent influence of resizing on flexural and tensile properties
- MF 43: resizing leads to decrease of strain / strength
- No influence of resizing and oxidation on modulus

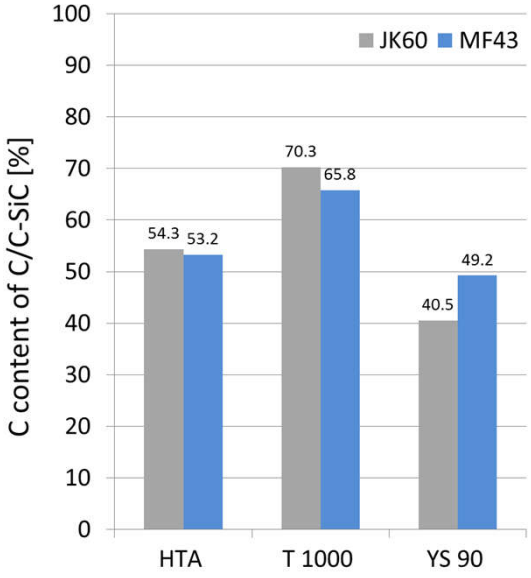


C/C-SiC Microstructures – YS 90

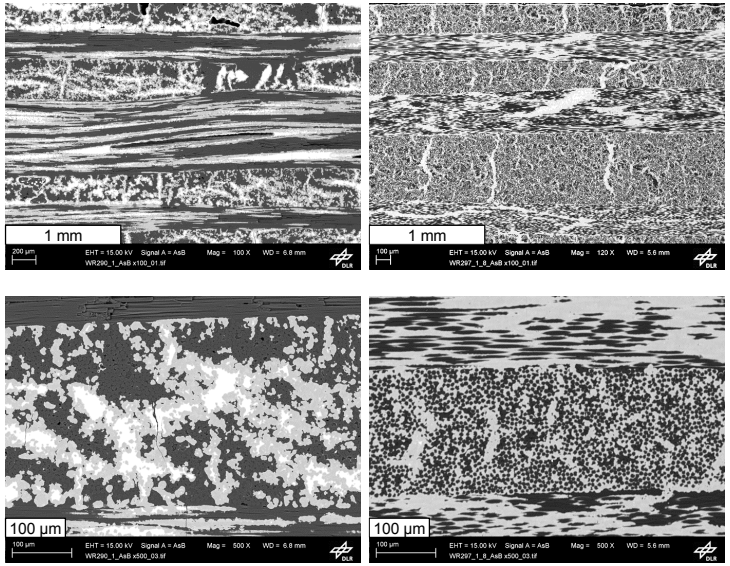
JK 60



HTA YS90



MF 43



HTA YS90

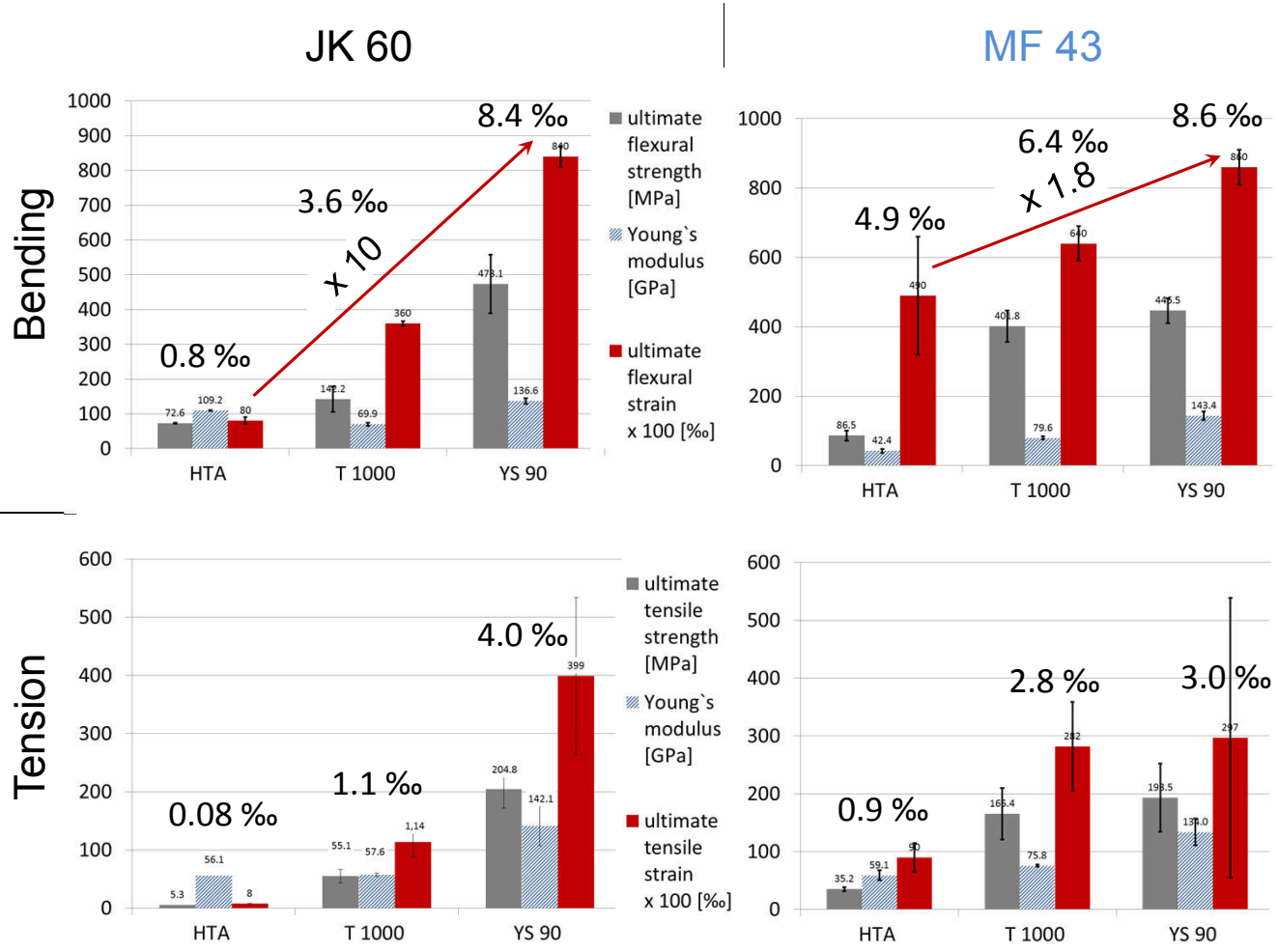
High conversion rate of YS90 based C/C-SiC and lowest C-content (40 / 49 %) of all material variants

Results – YS 90

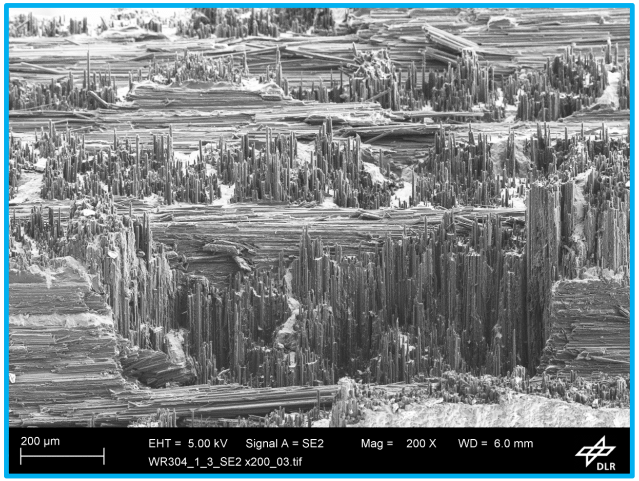
- YS 90 leads to highest strain and strength:

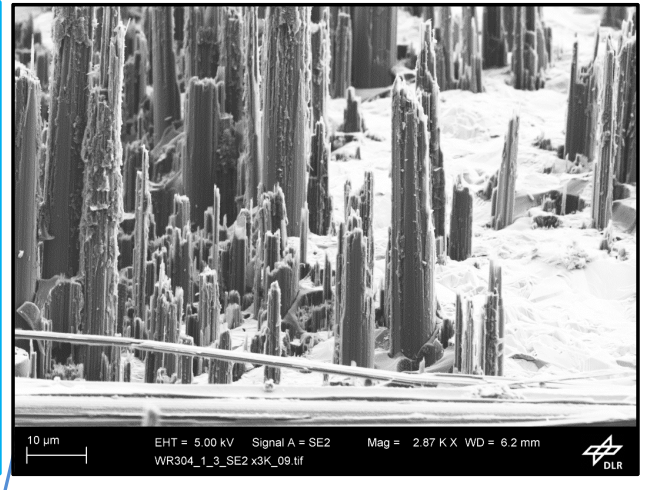
  ε_t = 4.0 ‰; σ_t = 205 MPa; E_t = 142 GPa
  ε_b = 8.4 ‰; σ_b = 473 MPa; E_b = 137 GPa

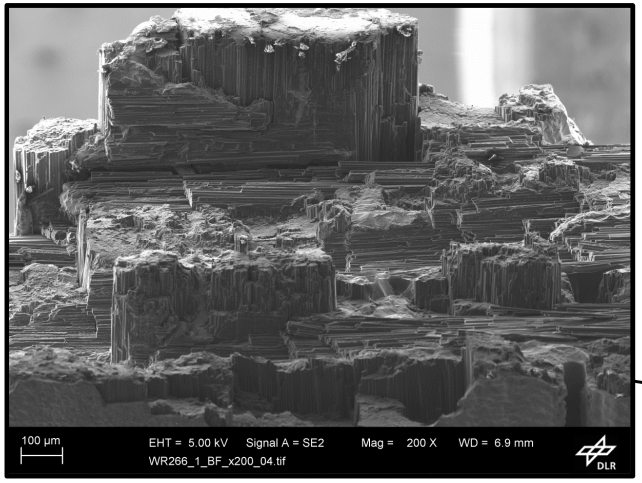
- Minor influence of precursor type

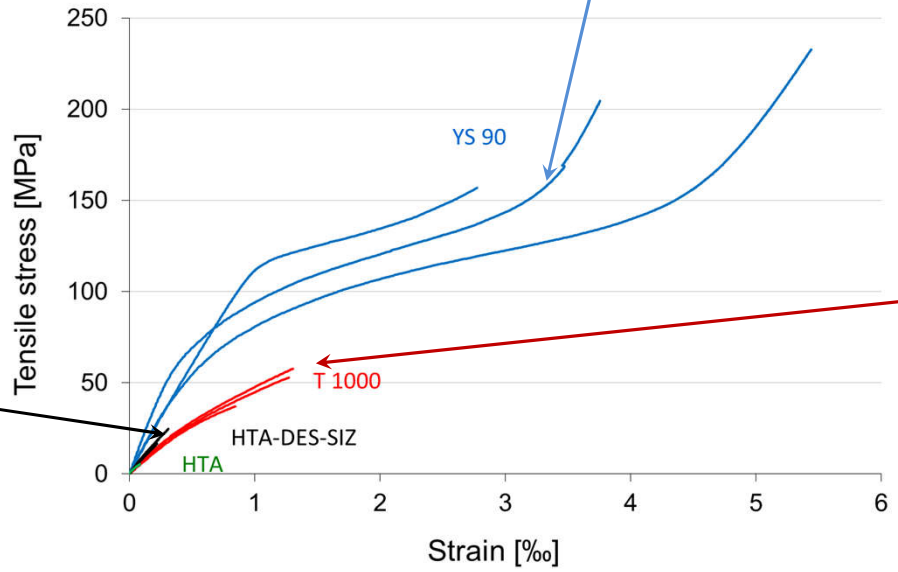


Fracture Behaviour







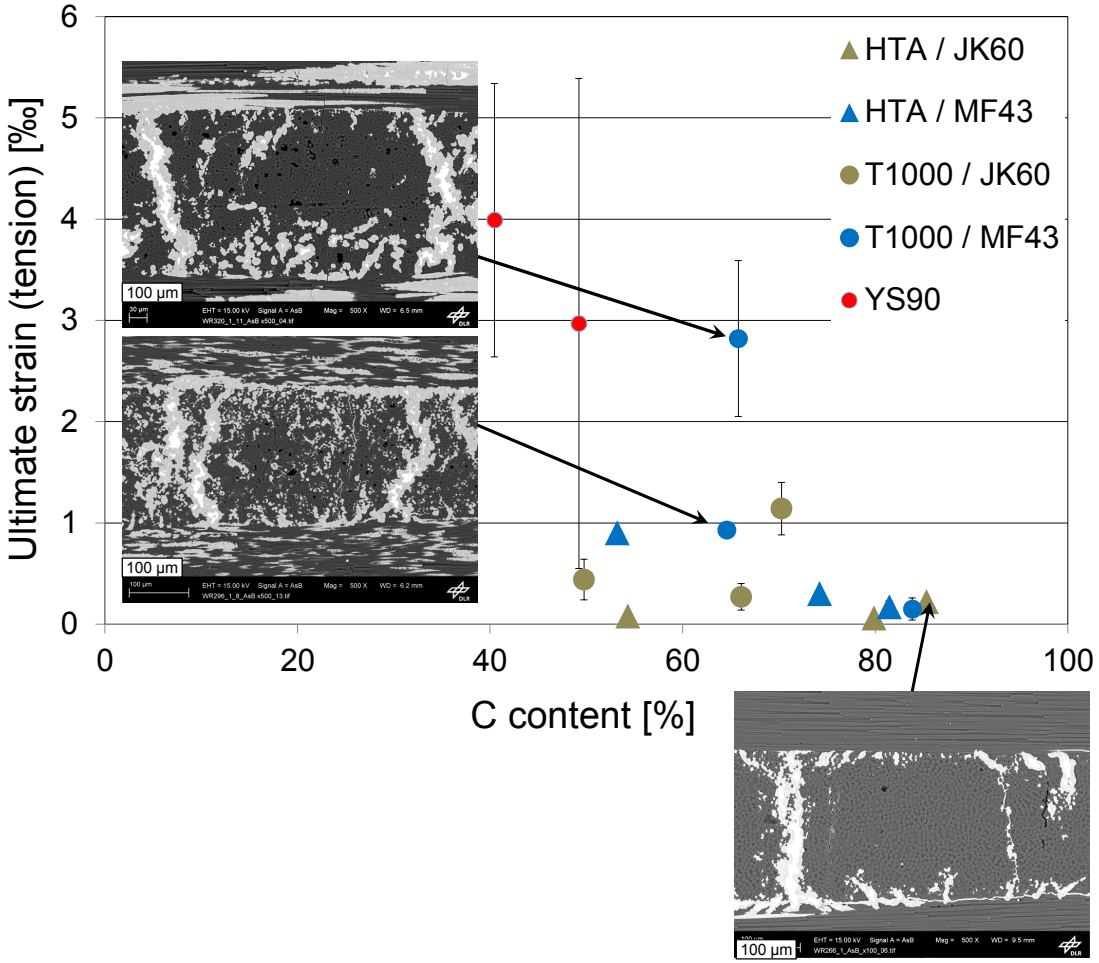


Influence of C-content on C/C-SiC properties

- Determination of C- content via grey scale analysis (ImageJ)
- No significant influence of C-content to fracture strain
- Absolute C-content not fully representative for defining the conversion rate

→ Focus on

- Distribution of C- or SiC content
- Bonding between fibers and C-Matrix
- Fracture behaviour of C/C bundles



Summary

- C/C-SiC with HT, UHT, UHM fibres (as delivered and modified) and two different phenolic precursors were manufactured via LSI and wet filament winding of CFRP preforms.
- Mechanical properties of C/C-SiC strongly influenced by matrix precursor and fibre type. High strain and strength obtained with:
  - Water based precursor with high C yield (MF43) (T1000: ε_t = 2.8 ‰; σ_t = 165 Mpa; E = 76 GPa) → preferred for wet filament winding
  - YS 90, due to “internal fibre pullout” (JK60: ε_t = 4.0 ‰; σ_t = 205 Mpa; E = 142 GPa).
- Resizing of HTA fibres prevents fibre matrix delamination (HTA / JK60)→ lower conversion rate → increased strength / strain (ε_t = 0.08 → 0.22 ‰)
- Microstructure analysis in C/C-SiC has to be adapted in order to define correlations with mechanical properties. Absolute phase contents → phase distribution

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