Mo- and W-Fiber Reinforced SiCN Ceramic Matrix Composites based on PIP process

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Overview

- Introduction and motivation
- Properties of Mo- and W-fibers
- Manufacture of Mo/SiCN and W/SiCN composites
- Mechanical properties of composites
- Microstructure and phase analysis of composites
- Thermodynamics and viable reactions during cristallisation
- First mechanical models of composites
- Summary and outlook

Introduction and motivation

- Monolithic ceramics are brittle, have high stiffness and low fracture strain, but show catastrophic failure when overloaded
- Ceramic fiber reinforced ceramic matrix composites show graceful failure when overloaded, but still have low fracture strain (compared to metals)
- Metal fiber reinforced ceramic matrix composites are very little known, however, could be interesting due to higher fracture strain of metallic fibers
- Ceramic matrices are more oxidation and corrosion resistant as well as light-weight compared to molybdenum and tungsten

Physical and mechanical properties of Mo- and W-fibers

Fiber type		**Tungsten** **BSD-OG-102045280100**	**Molybdenum** **MOA-B6144601XX42**
Manufacturer		Osram	Osram
Diameter	µm	150	200
Density	g/cm³	19.250	10.220
Yield strength	MPa		1207±5
Tensile strength	MPa	2774±29 [Riesch2017]	1647±1
Tensile modulus	GPa		287±2
Fracture strain	%		1.9±0.1
Reduction in area	%	37±3 [Riesch2017]	70.2±0.2
K content	ppm	70-80	150-200

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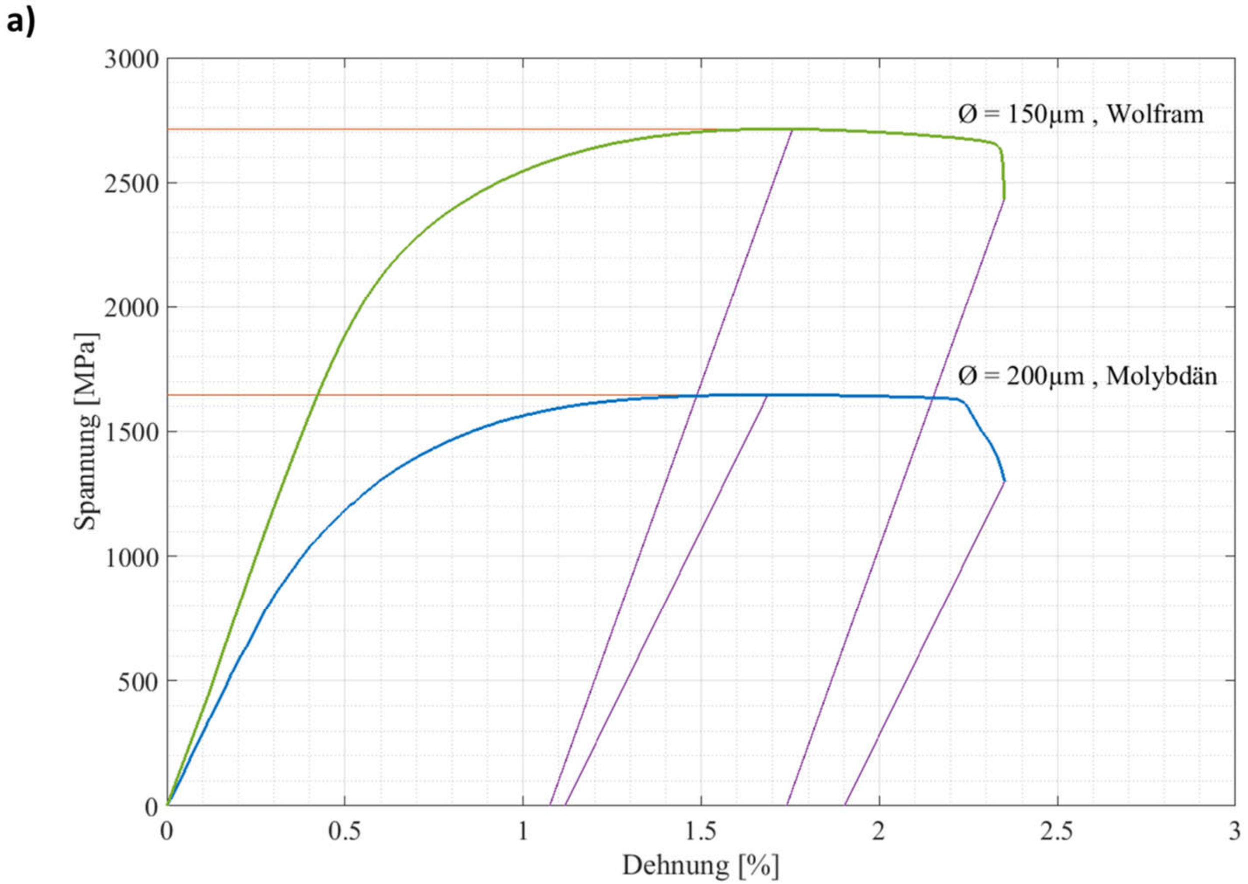
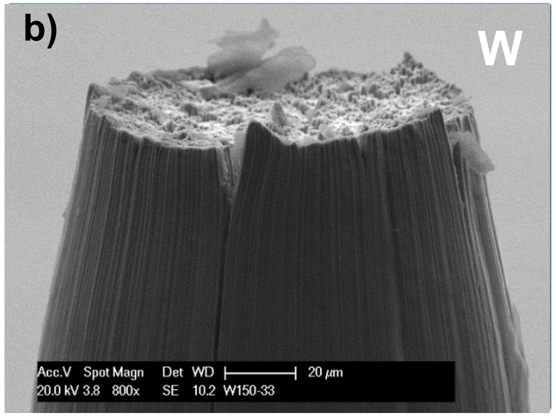
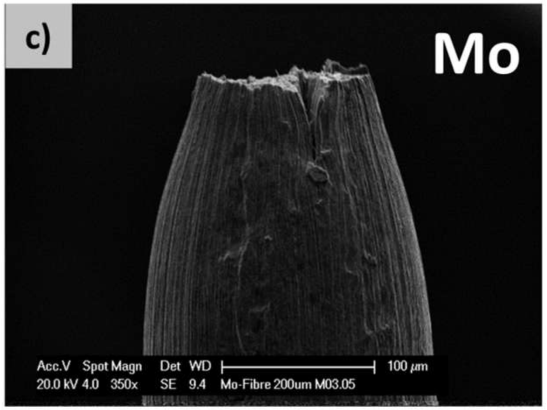
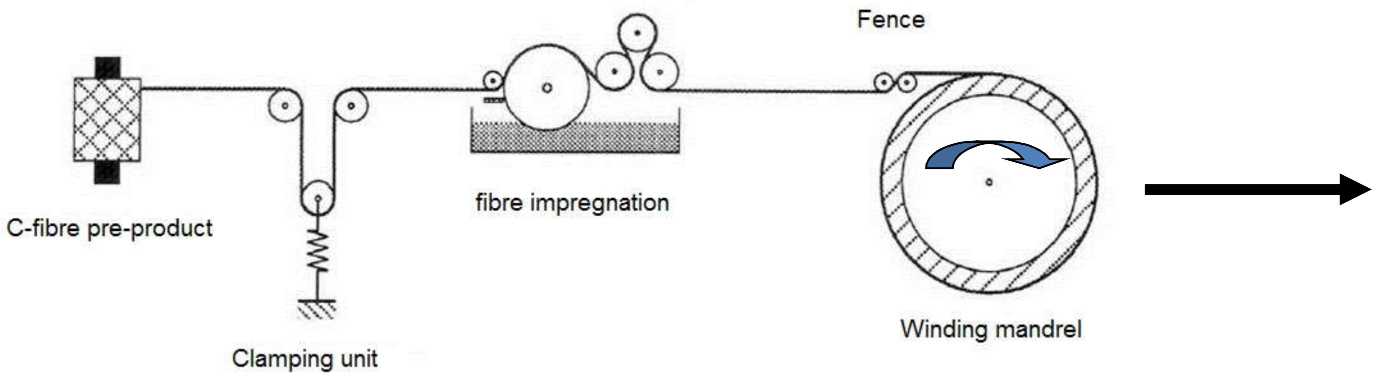
Tensile testing of single Mo- and W-fibers

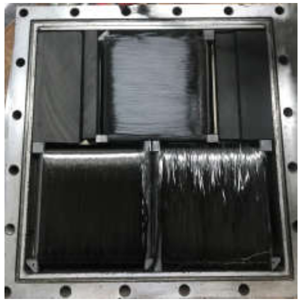


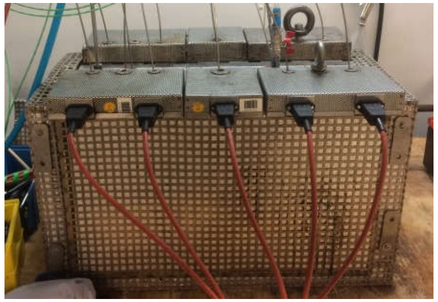




Preform Manufacture – Dry Filament Winding



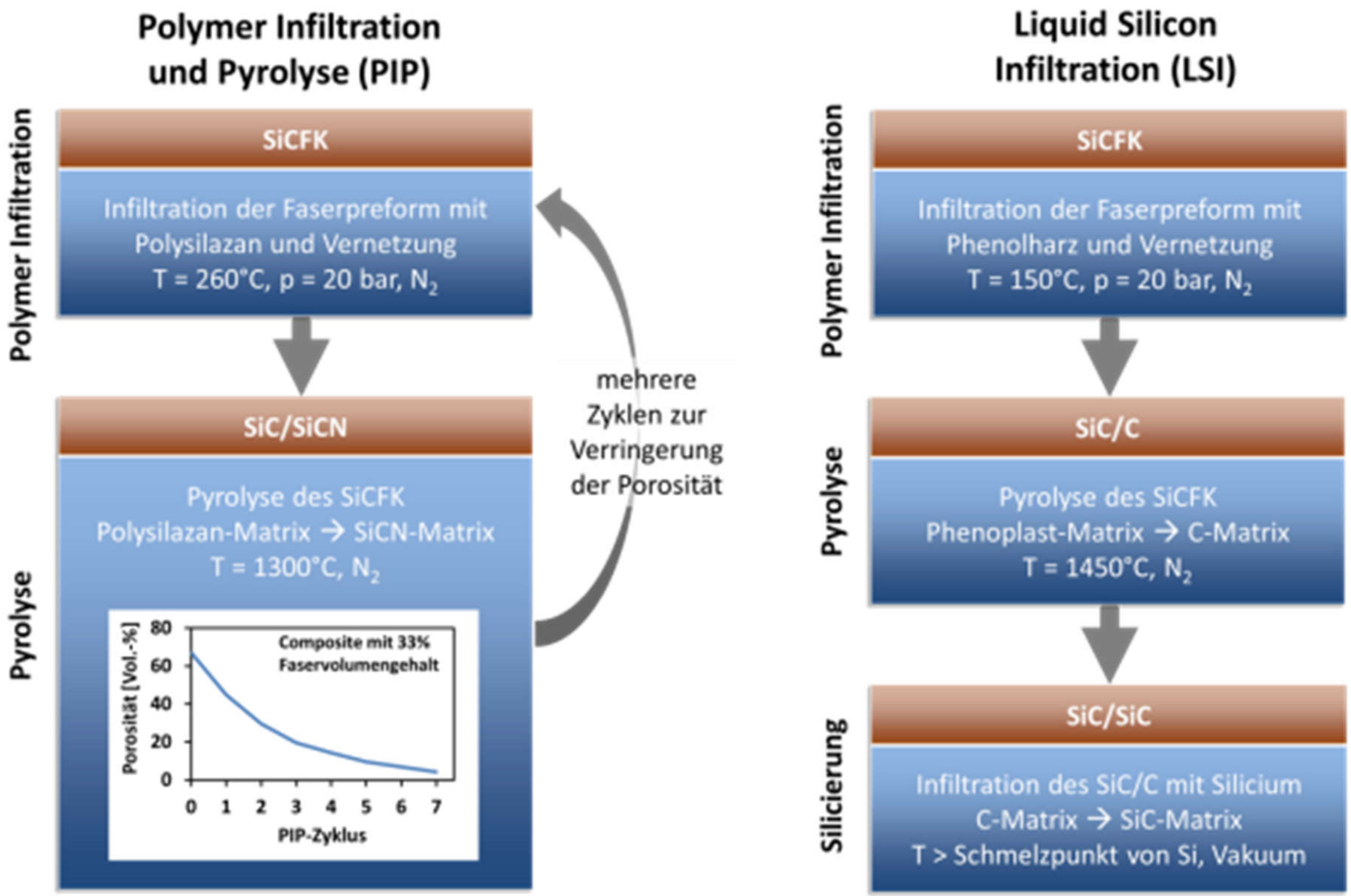




Raw materials and equipment:

- Mo- or W-fibers
- Filament winding machine controlling winding speed and angle
- Graphite mandrel equipped with Teflon tape
- Precursor PSZ10 (polysilazane resin) for RTM infiltration
- Steel mould for RTM infiltration and curing under pressure

Manufacture of Mo- and W-fiber ceramic matrix composites



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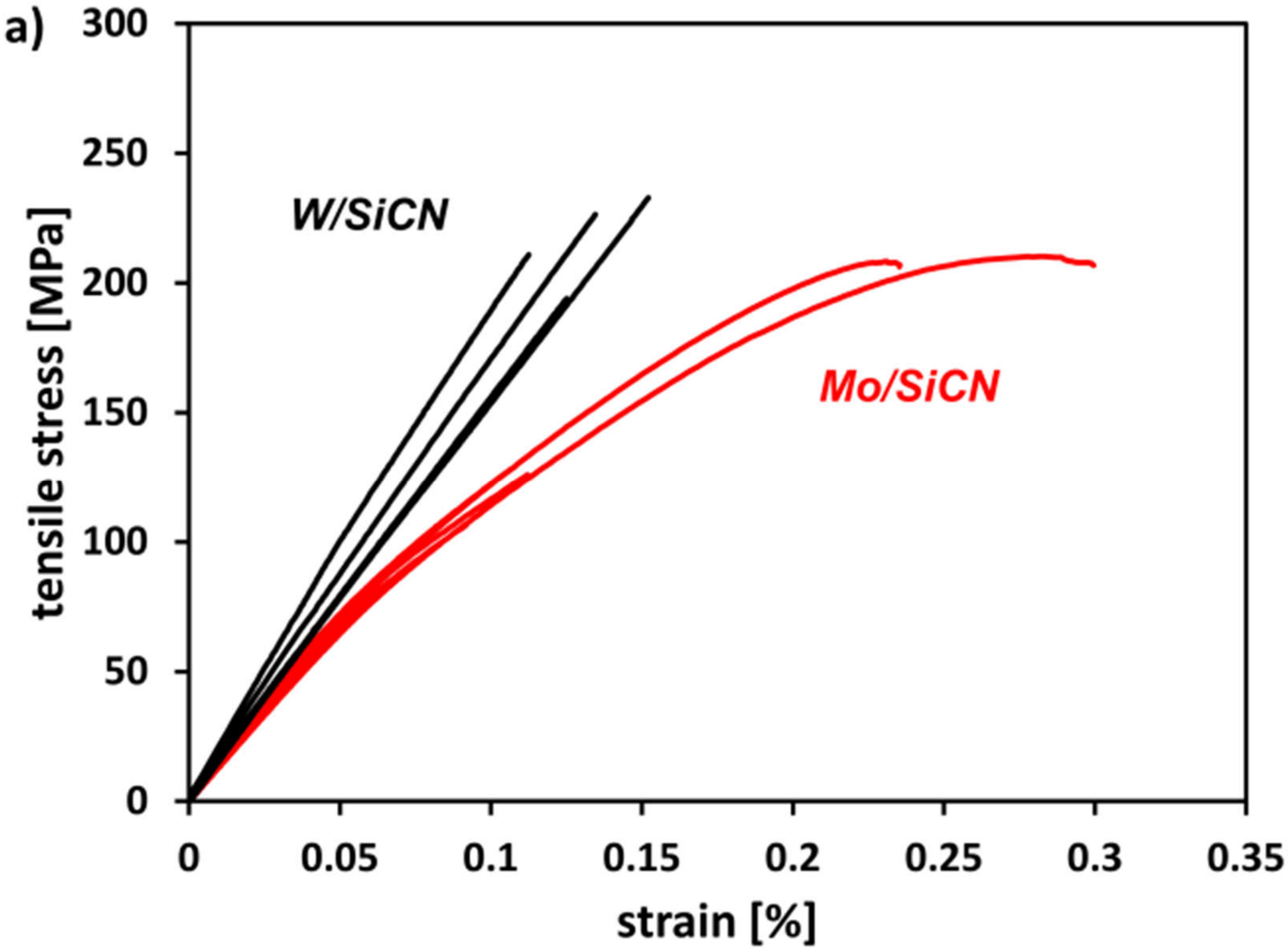
Properties of Mo/SiCN and W/SiCN composites

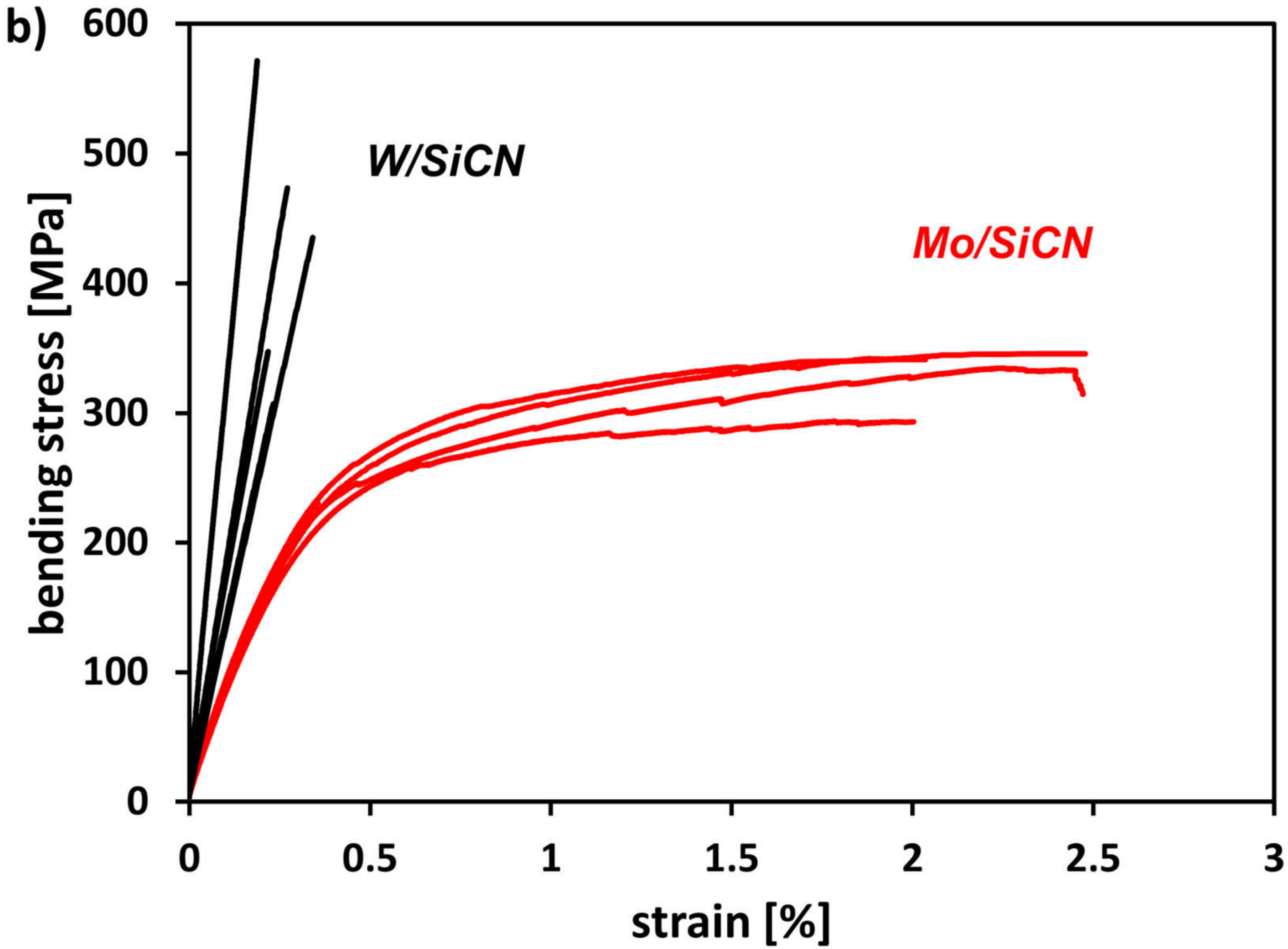
Composite type		**W/SiCN**	**Mo/SiCN**
Fiber volume content	%	25 (33*)	30
Tensile strength	MPa	206±27	156±50
Tensile modulus	GPa	172±19	144±7
Tensile fracture strain	%	0.126±0.018	0.164±0.086
Bending strength	MPa	427±105	312±50
Bending modulus	GPa	193±89	90±6
Bending fracture strain	%	0.24±0.08	2.02±0.93
Density	g/cm^3	7.72	4.44
Porosity	Vol.-%	6.86	10.07
Density (calculated)	g/cm^3	6.38 (7.74)	4.44

*calculated by asuming 2.30 g/cm^3 for density of SiCN

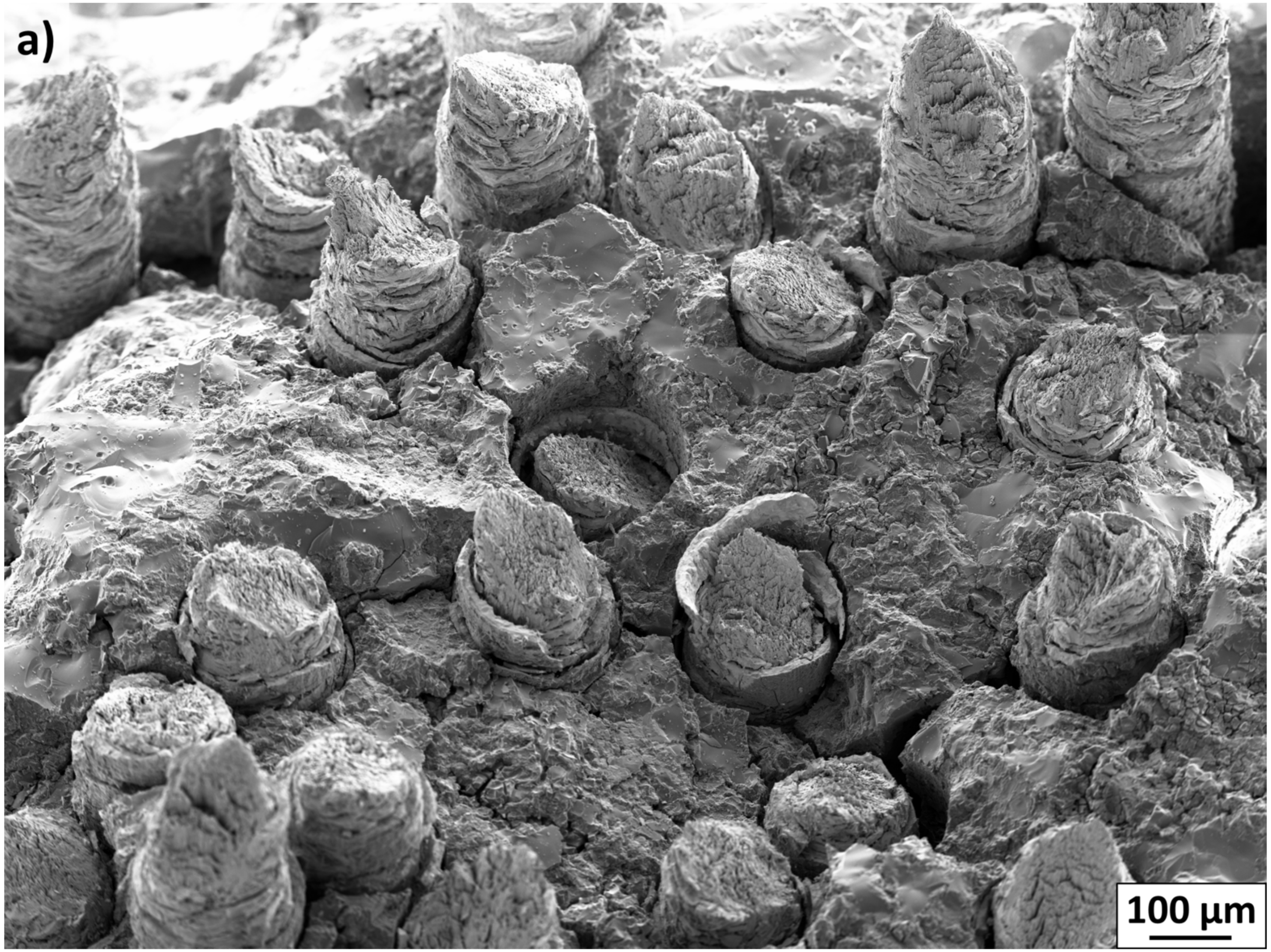
Tensile testing of Mo/SiCN and W/SiCN



Bending testing of Mo/SiCN and W/SiCN

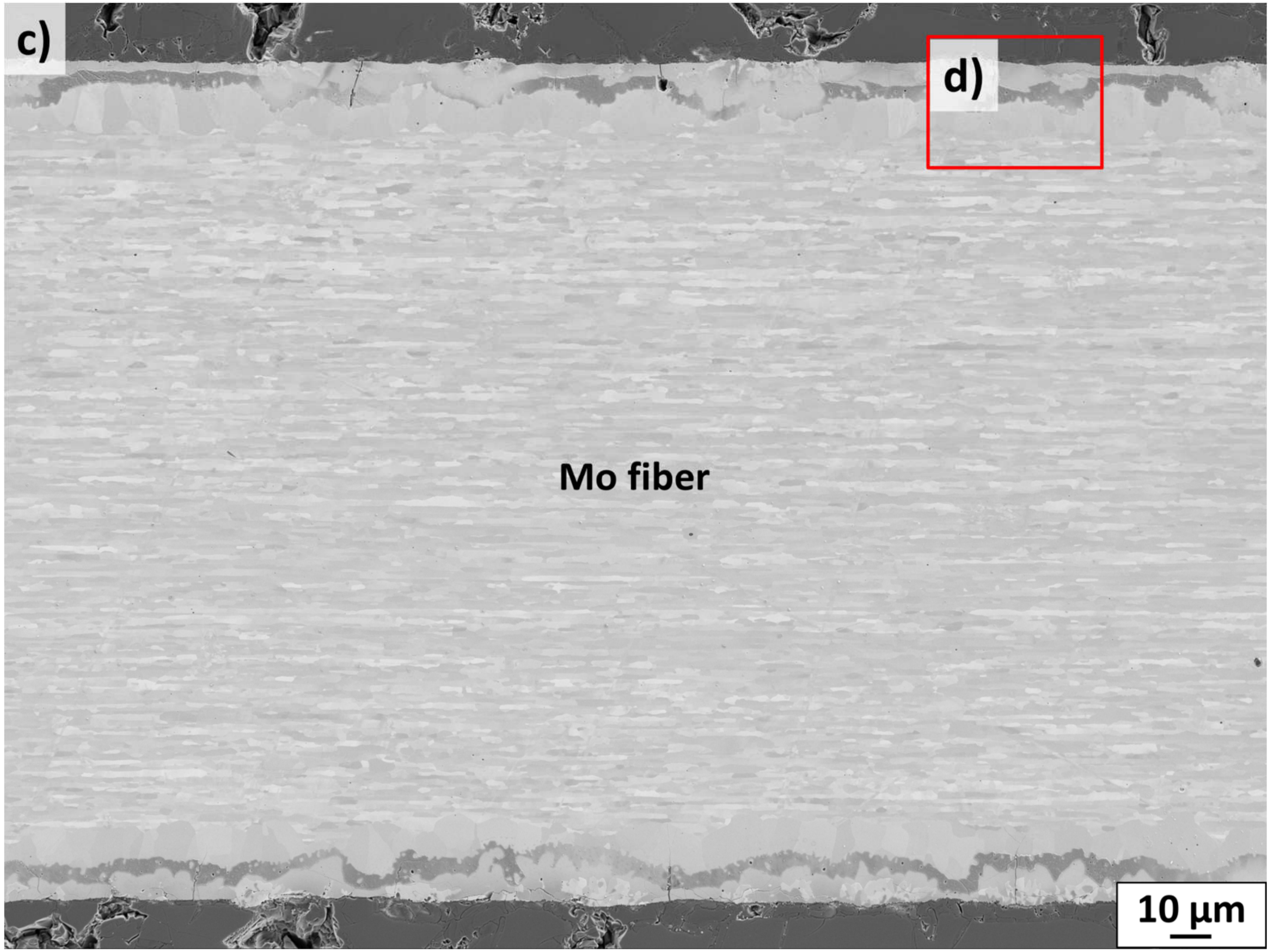


Microstructure of Mo/SiCN (I)

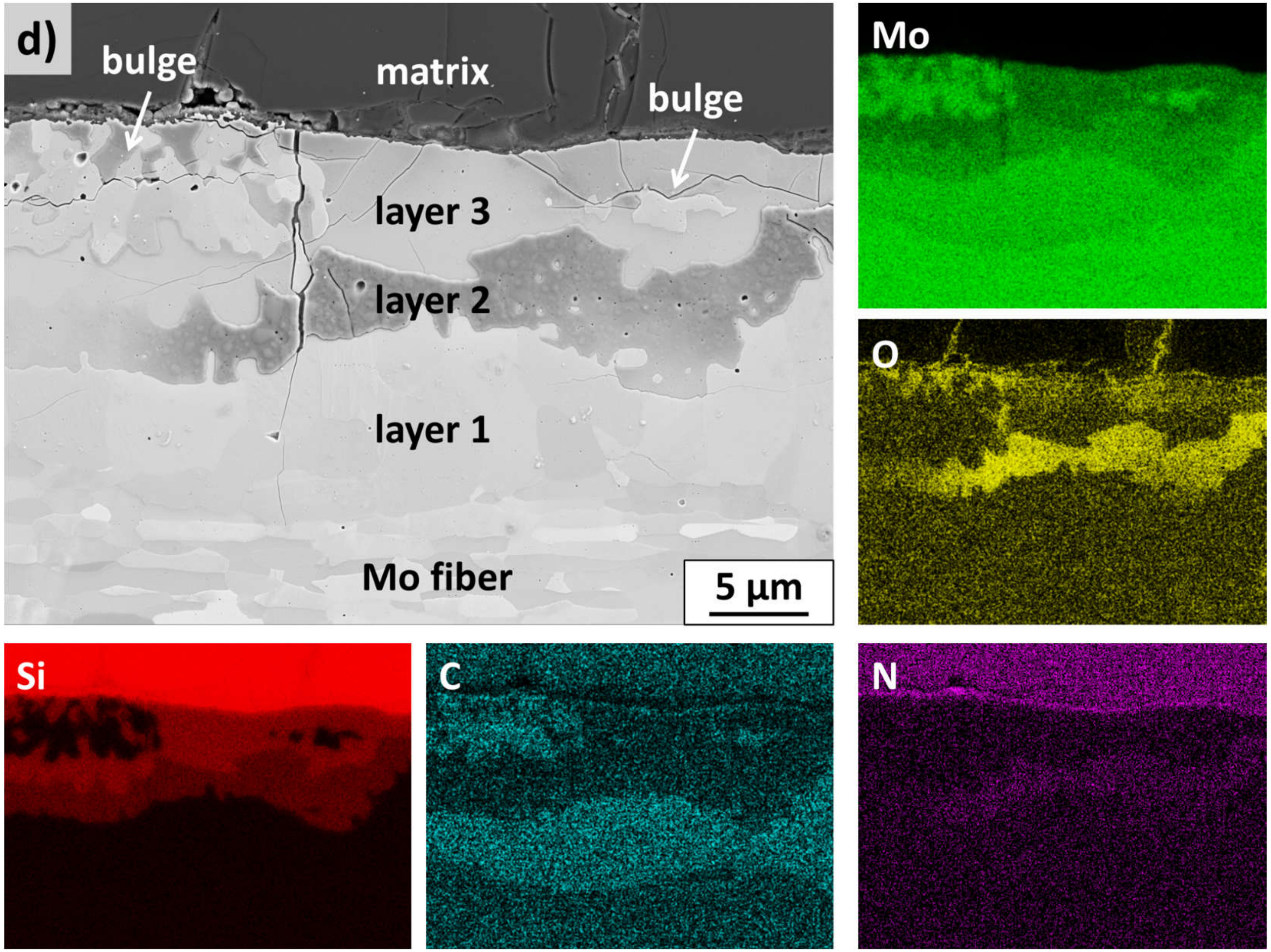


Microstructure of Mo/SiCN (II)

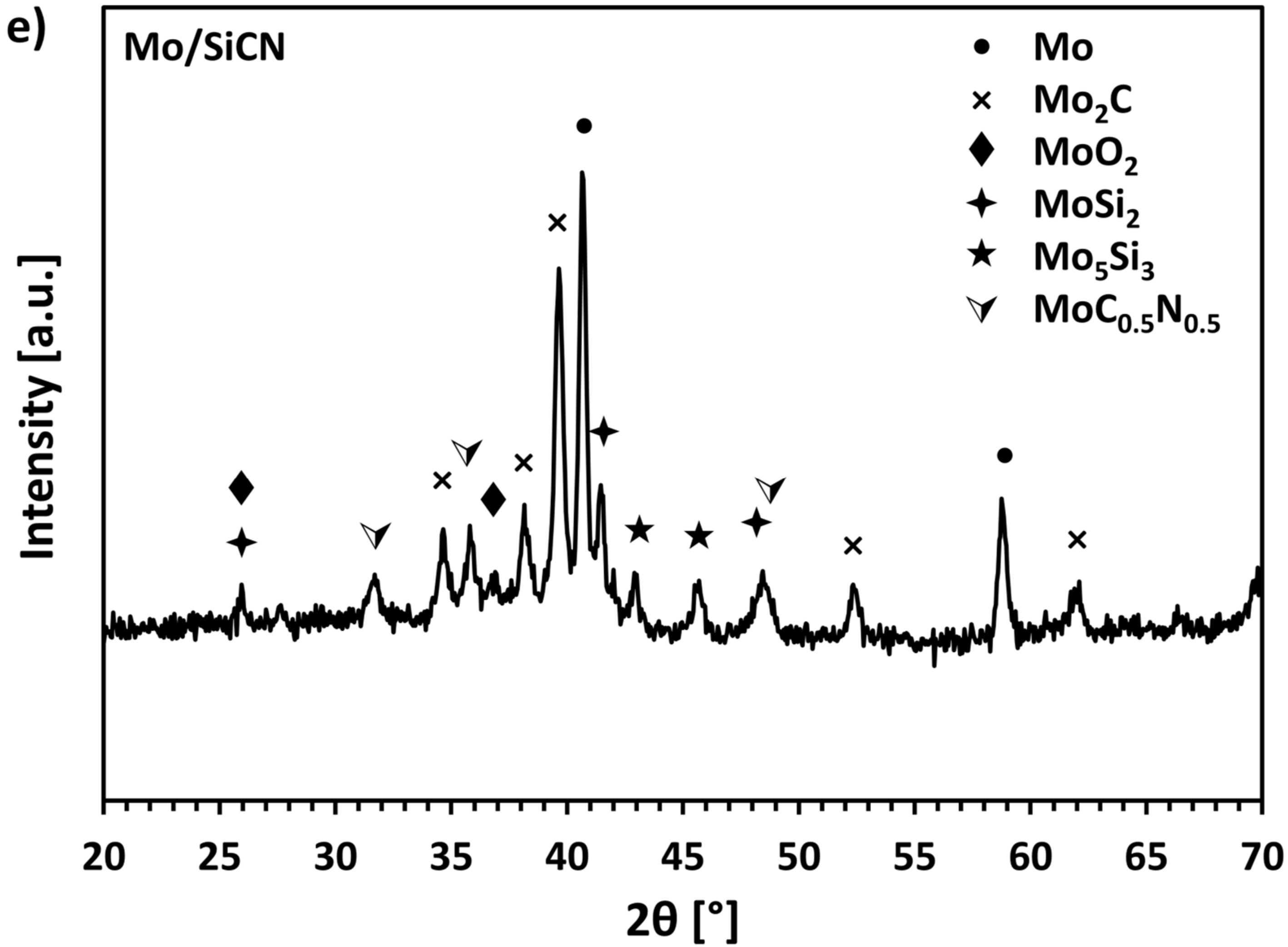
Microstructure of Mo/SiCN (III)



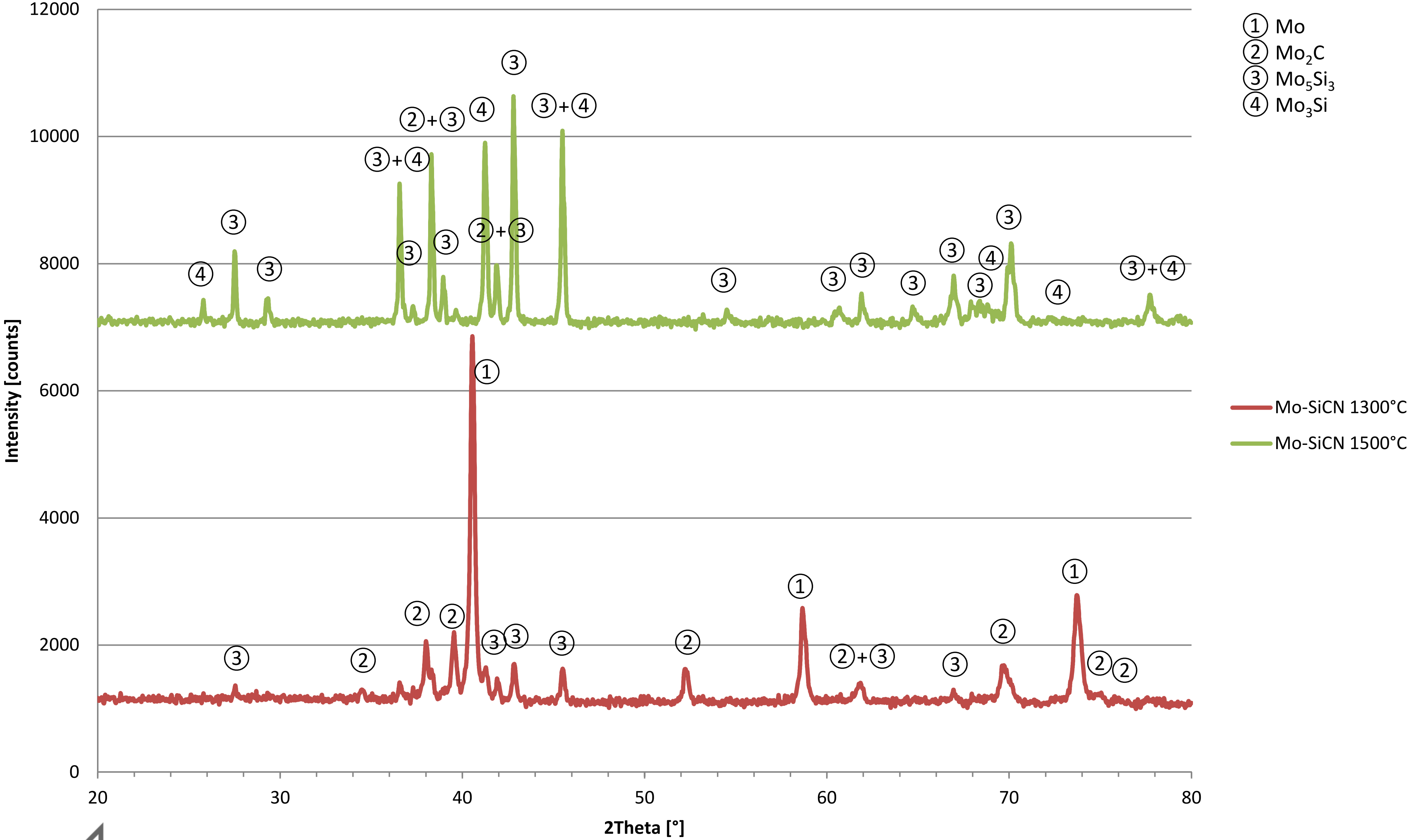
Microstructure and EDX-analysis of Mo/SiCN



First XRD-analysis of Mo/SiCN



High-res investigation on crystallisation of Mo/SiCN



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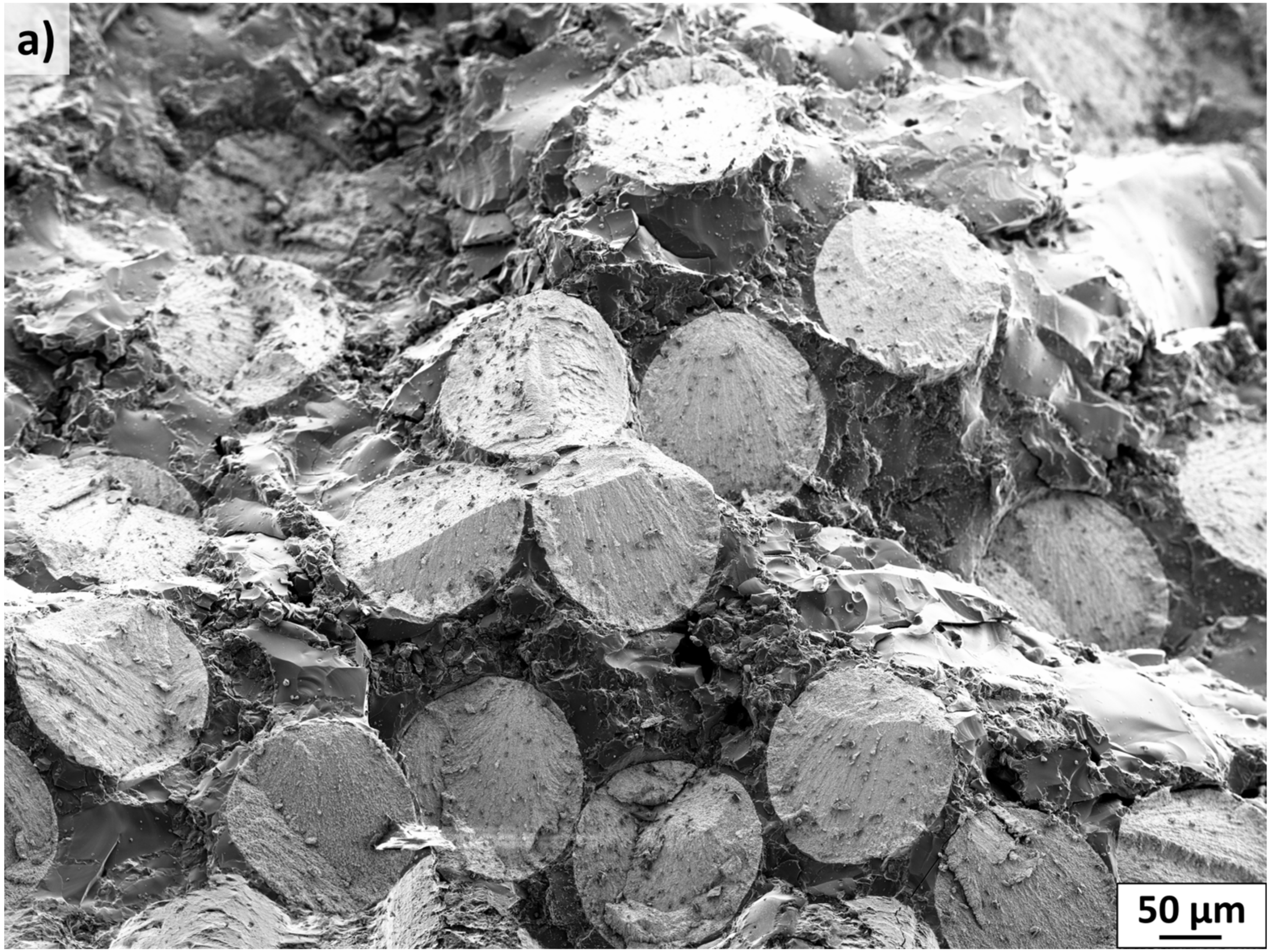
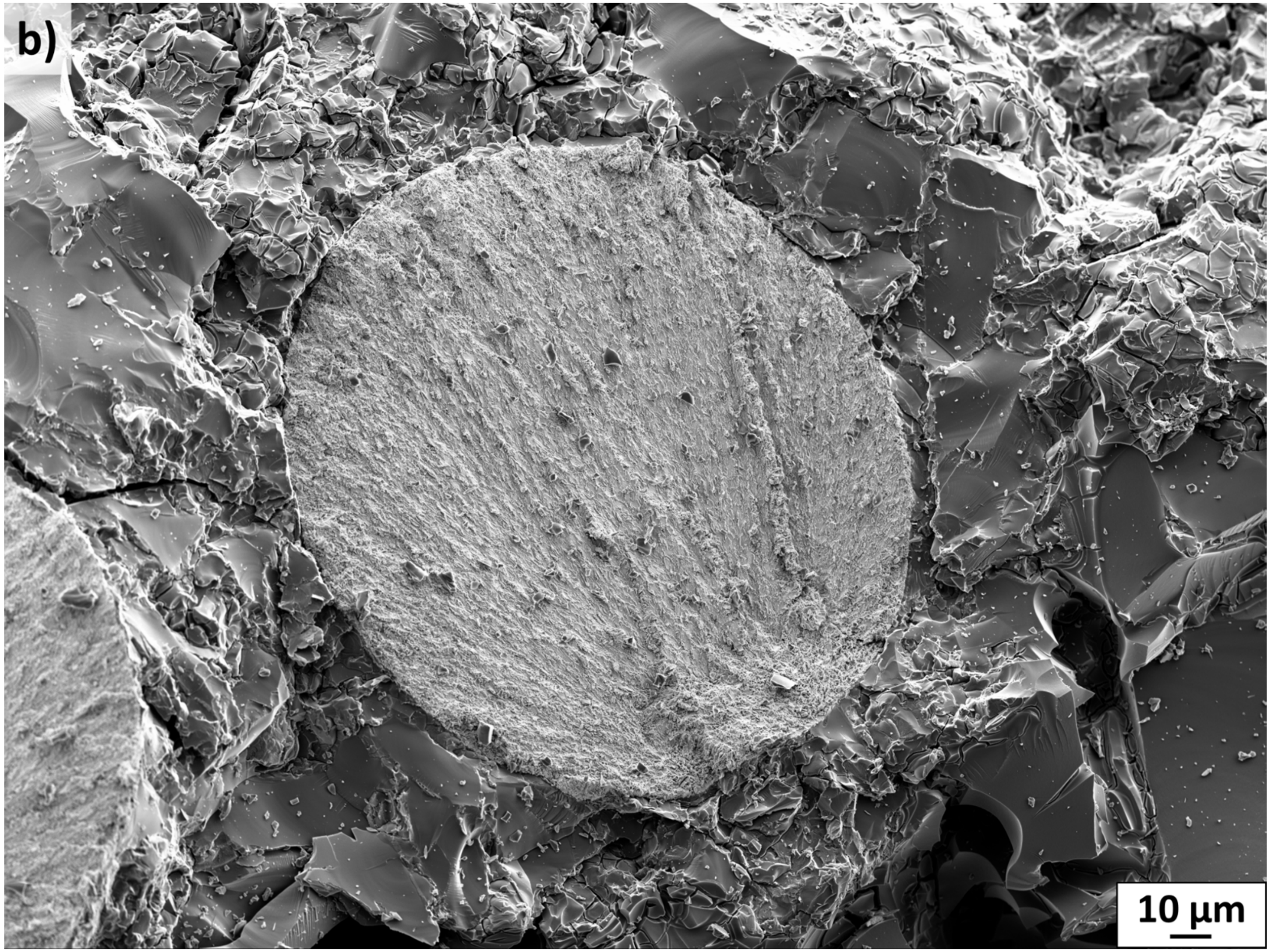
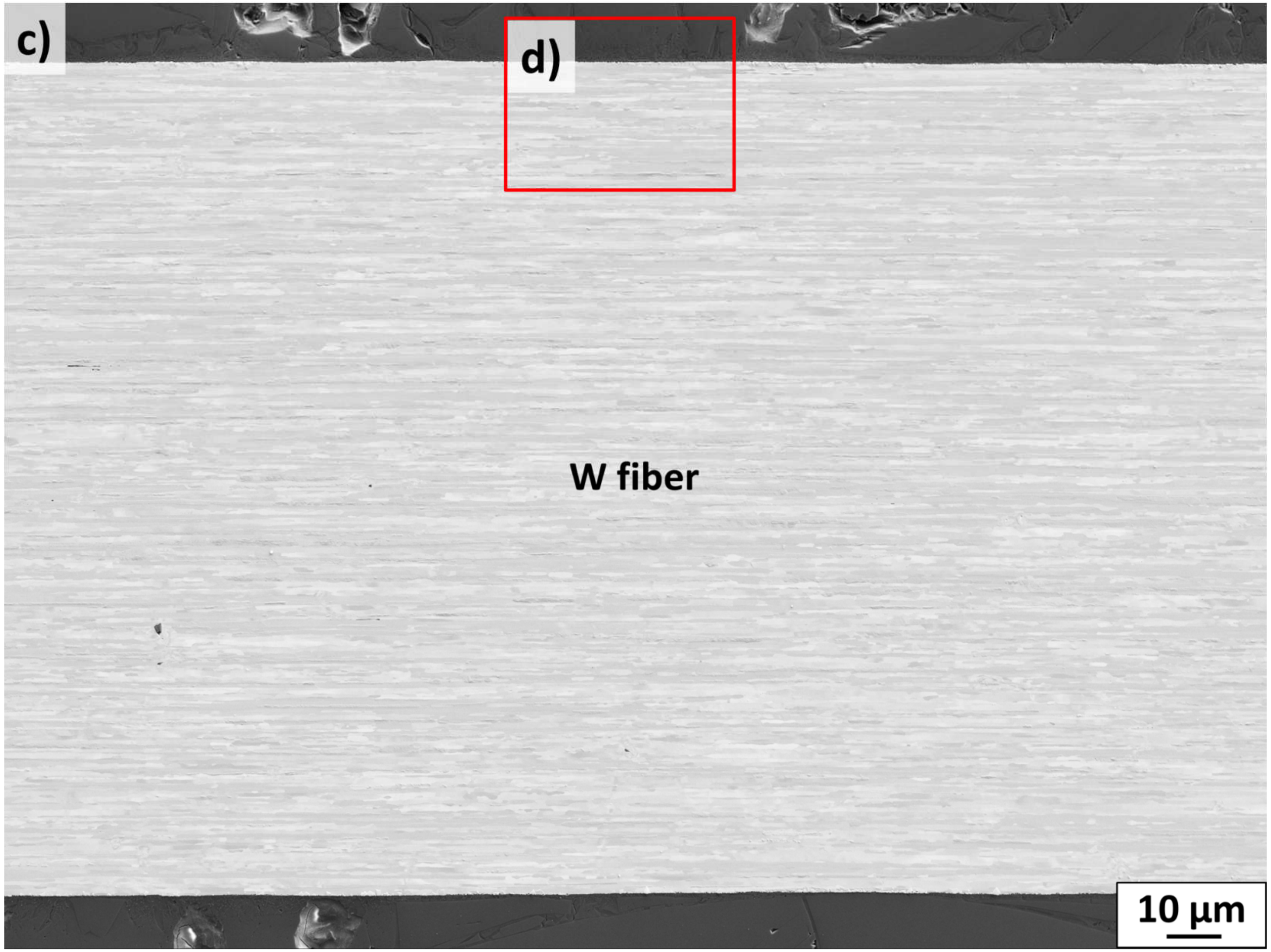
Microstructure of W/SiCN (I)



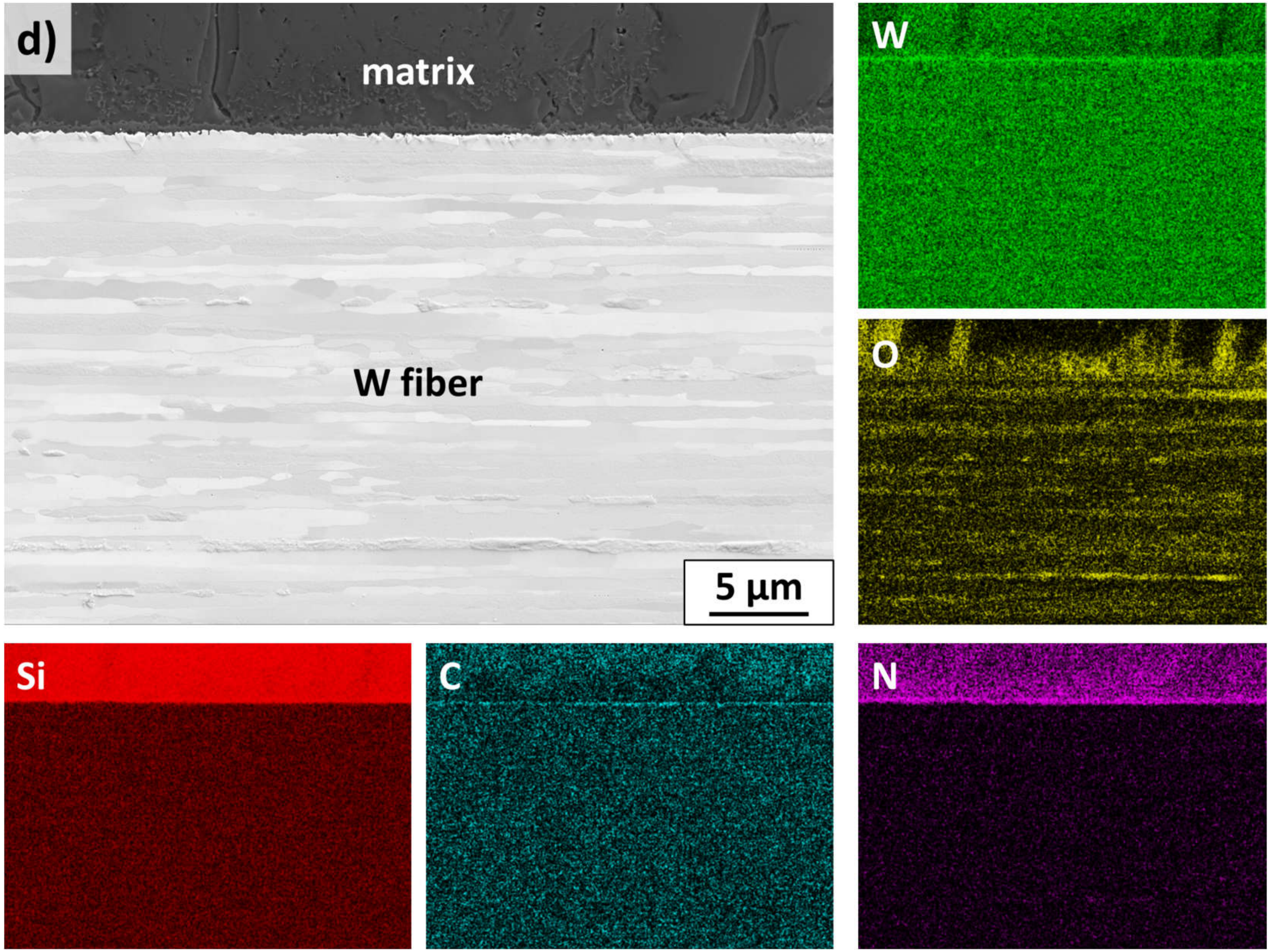
Microstructure of W/SiCN (II)



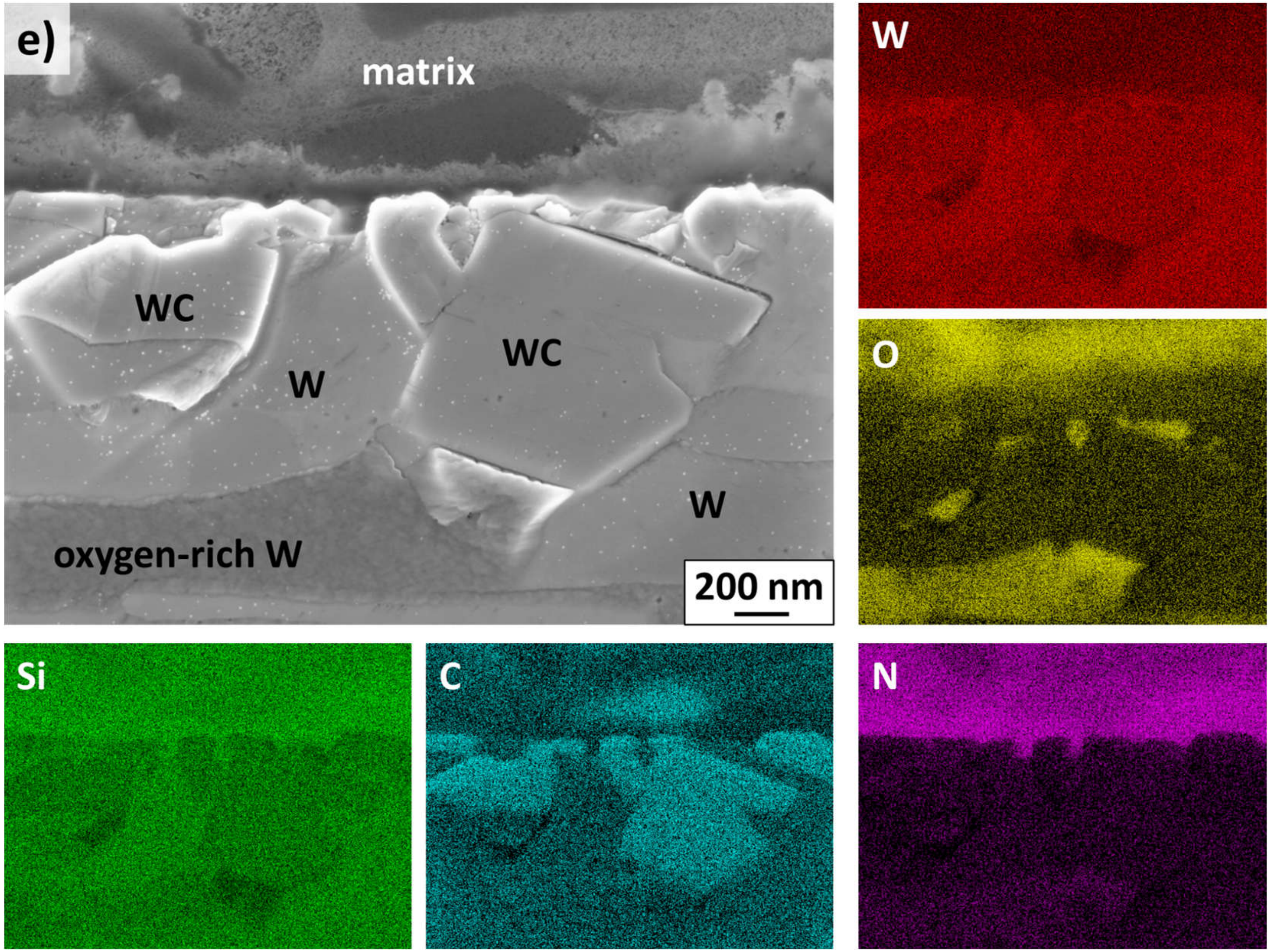
Microstructure of W/SiCN (III)



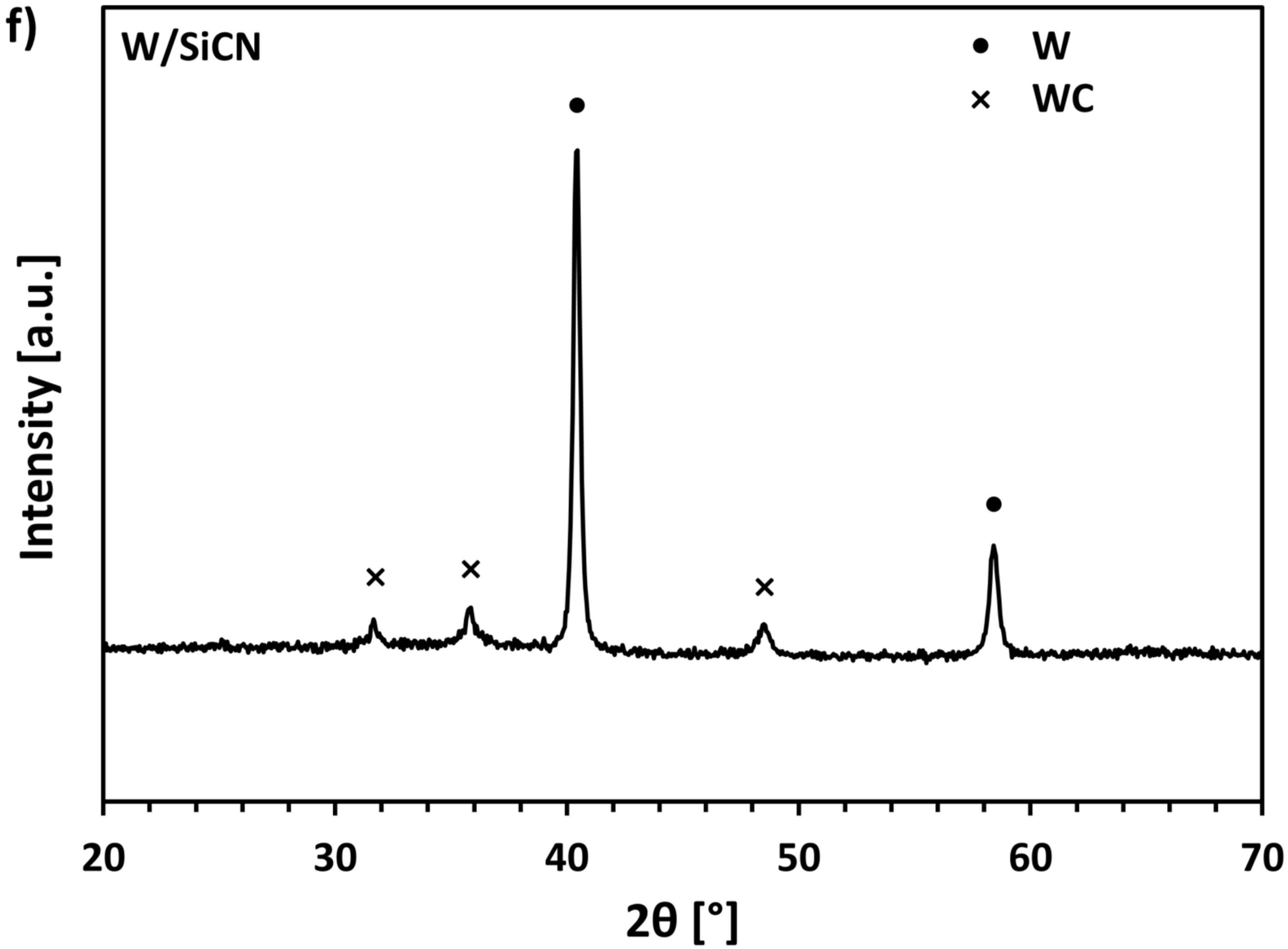
Microstructure and EDX-analysis of W/SiCN (I)



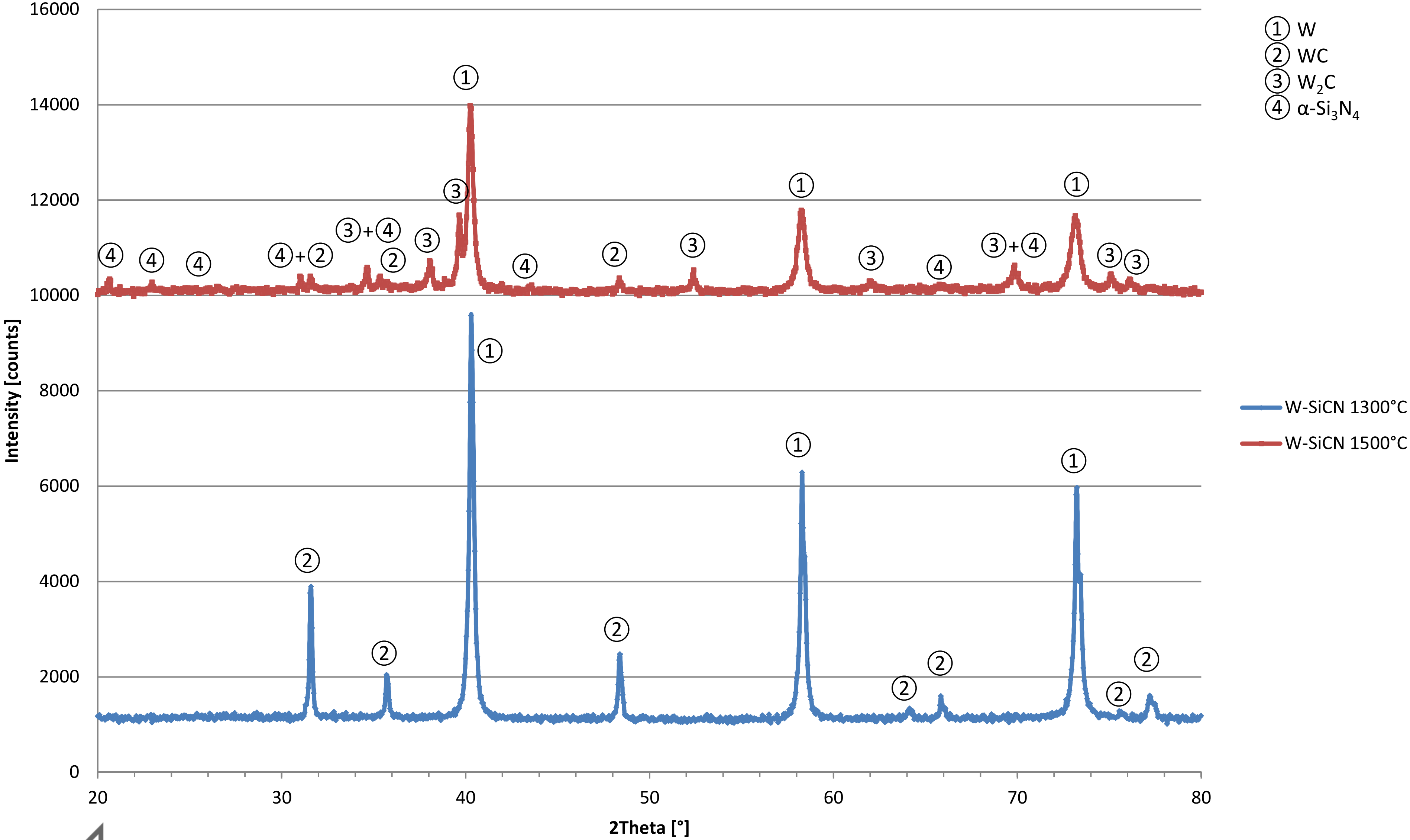
Microstructure and EDX-analysis of W/SiCN (II)



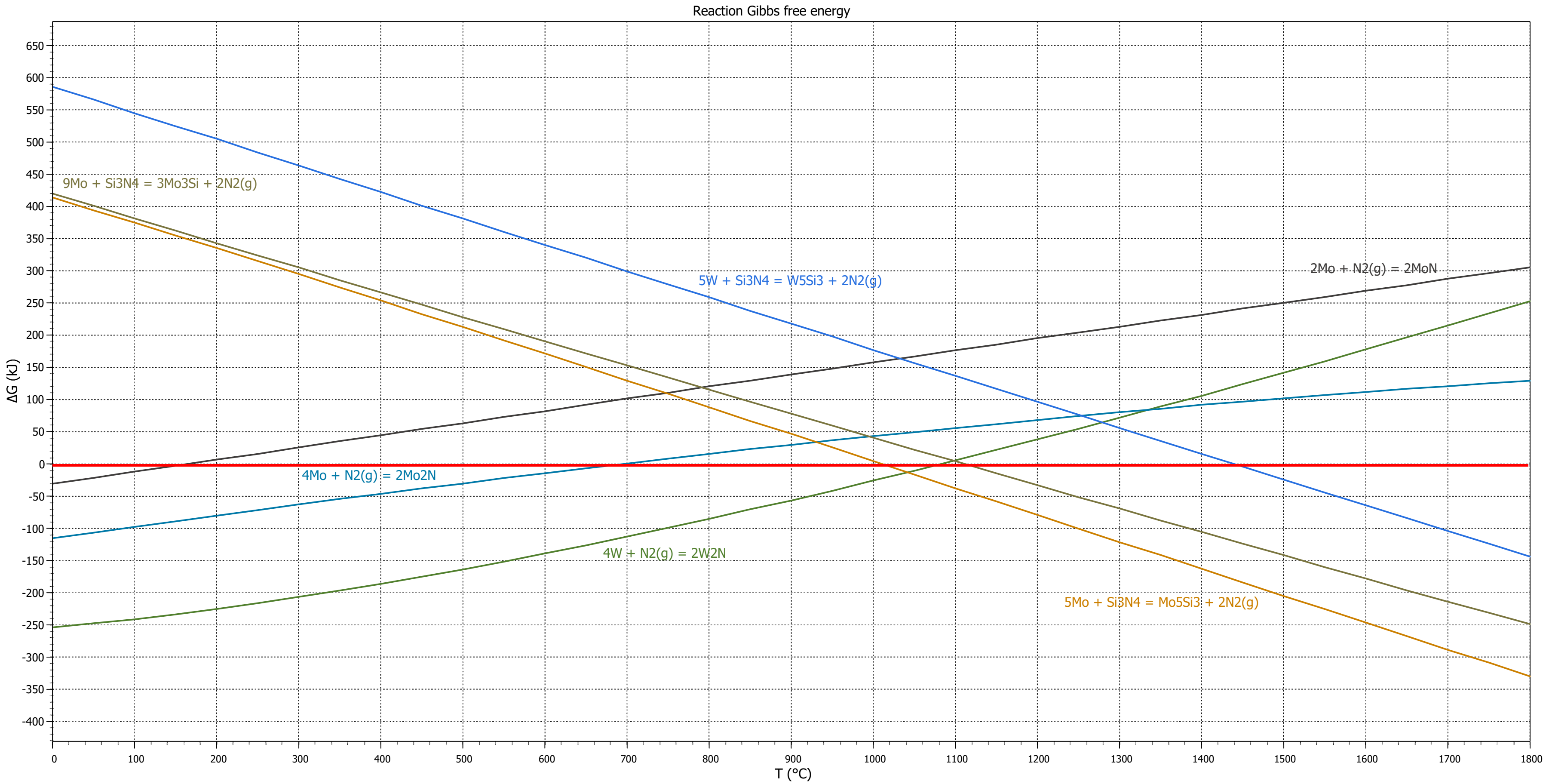
First XRD-analysis of W/SiCN



High-res investigation on crystallisation of W/SiCN

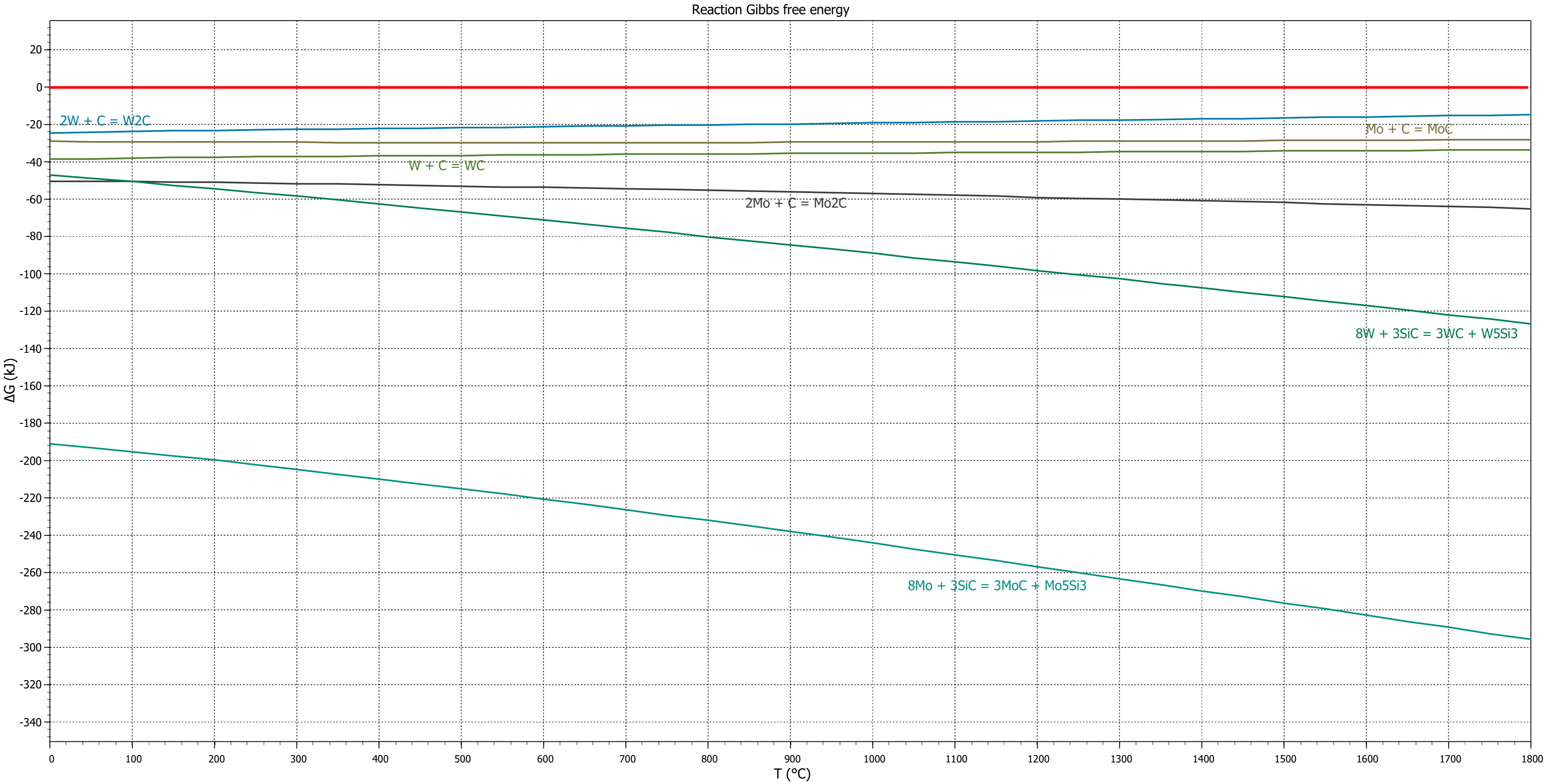


Thermodynamics: reactions of W or Mo with Si_3N_4



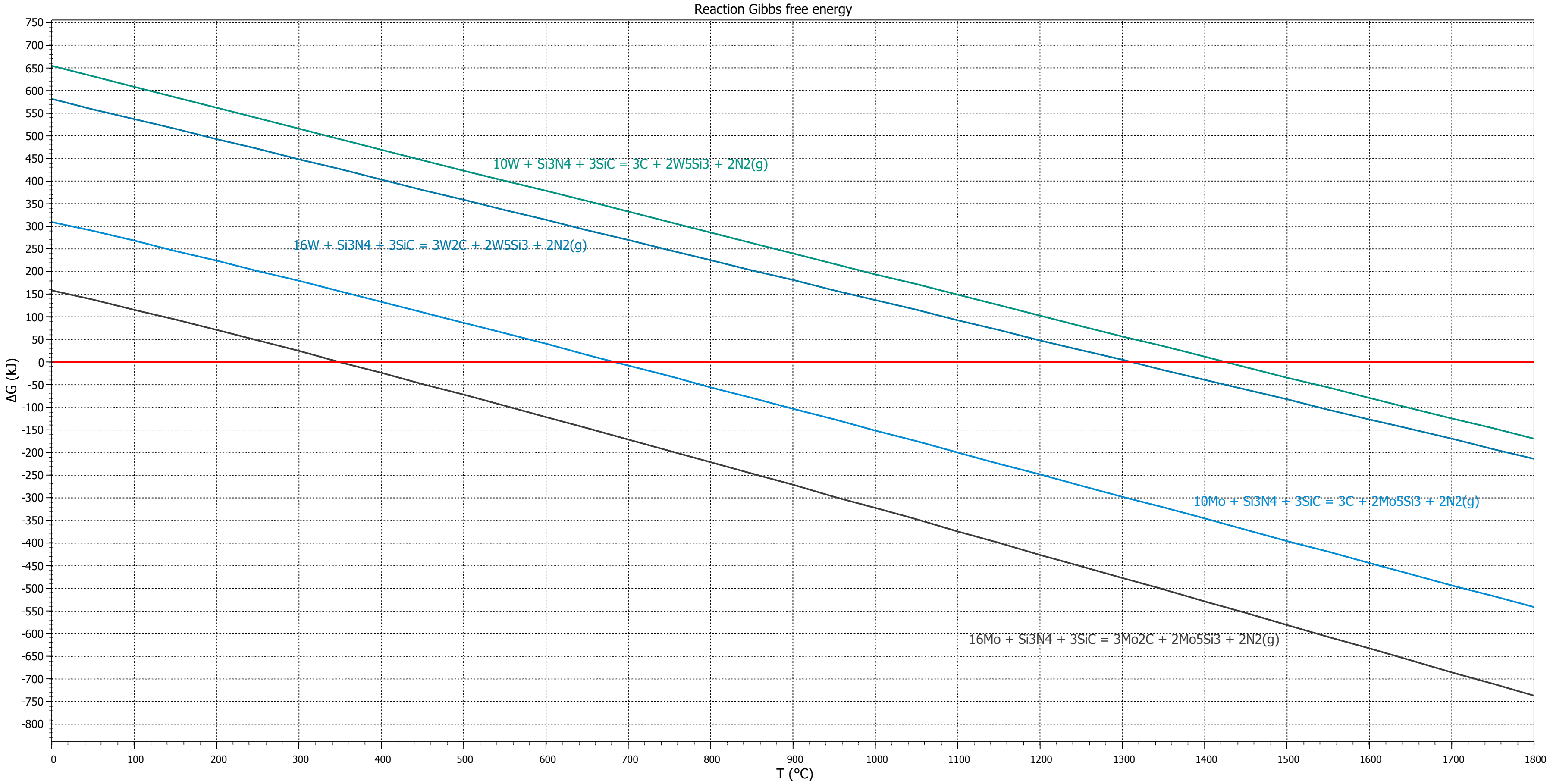
→ Reactions of N_2 with Mo or W are neither favoured thermodynamically nor kinetically
→ Reactions under N_2 release are preferred

Reactions of W or Mo with SiC

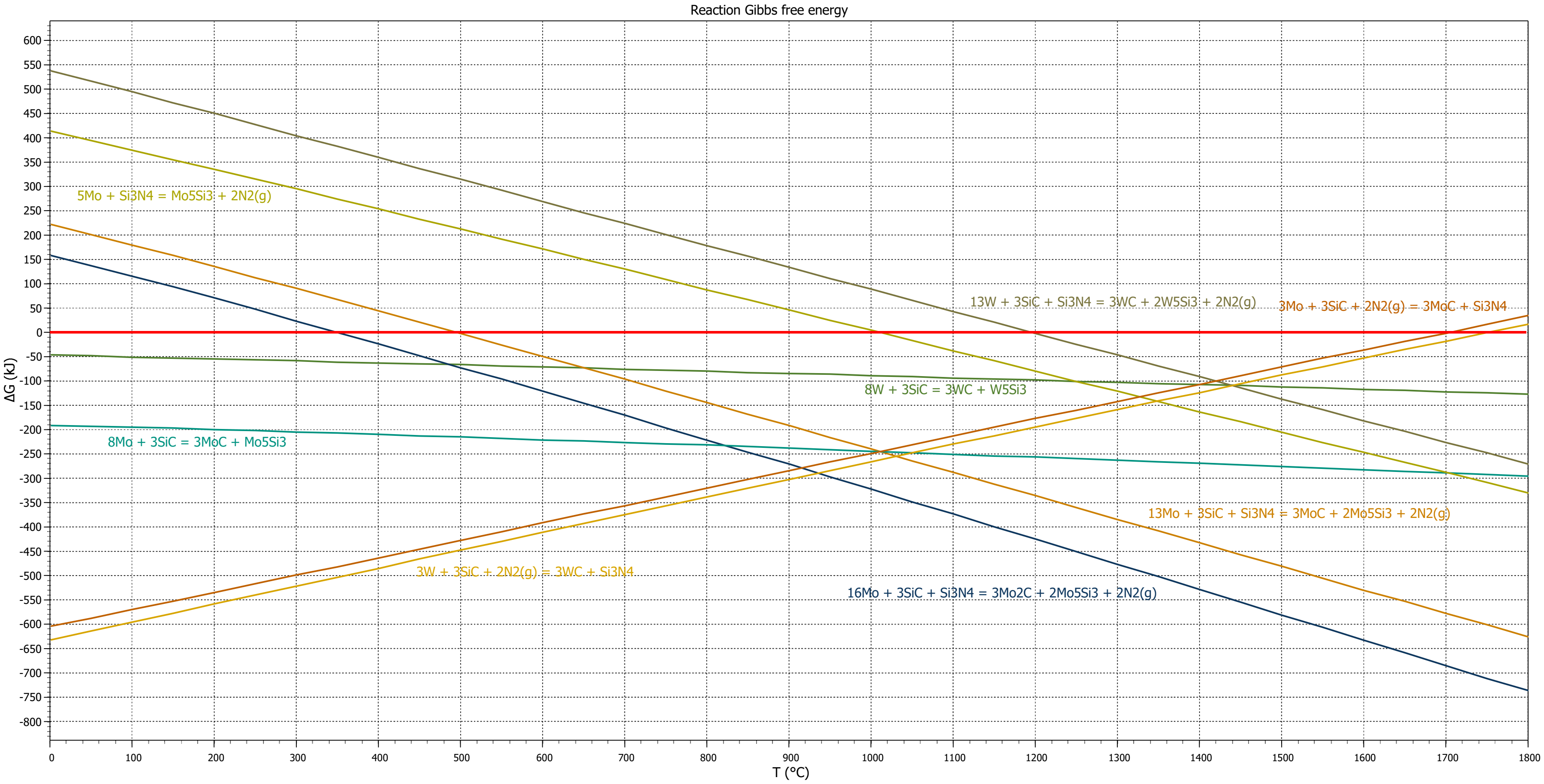


→ Reactions of Mo and W with C-compounds are preferred

Reactions of W or Mo with SiC and Si_3N_4



Preference of reactions of W and Mo



Viable reactions of Mo and W with SiCN w.r.t. TG-analysis

- **Mo + SiCN $\rightarrow$1300°C$\rightarrow$ $Mo + Mo_5Si_3 + Mo_2C + N_2\uparrow$**

  $4Mo + Mo_5Si_3 \leftrightarrow 3Mo_3Si$

  $5Mo + Si_3N_4 \leftrightarrow Mo_5Si_3 + 2N_2\uparrow$ **(mass loss! TG ok)**

- **Mo + SiCN $\rightarrow$1500°C$\rightarrow$ $Mo_5Si_3 + Mo_3Si + Mo_2C + N_2\uparrow$**

- **W + SiCN $\rightarrow$1300°C$\rightarrow$ $W + WC + SiCN + N_2\uparrow$**

  $W + WC \leftrightarrow W_2C$

  $3W + 3SiC + 2N_2 \leftrightarrow 3WC + Si_3N_4$ **(mass gain! TG ok)**

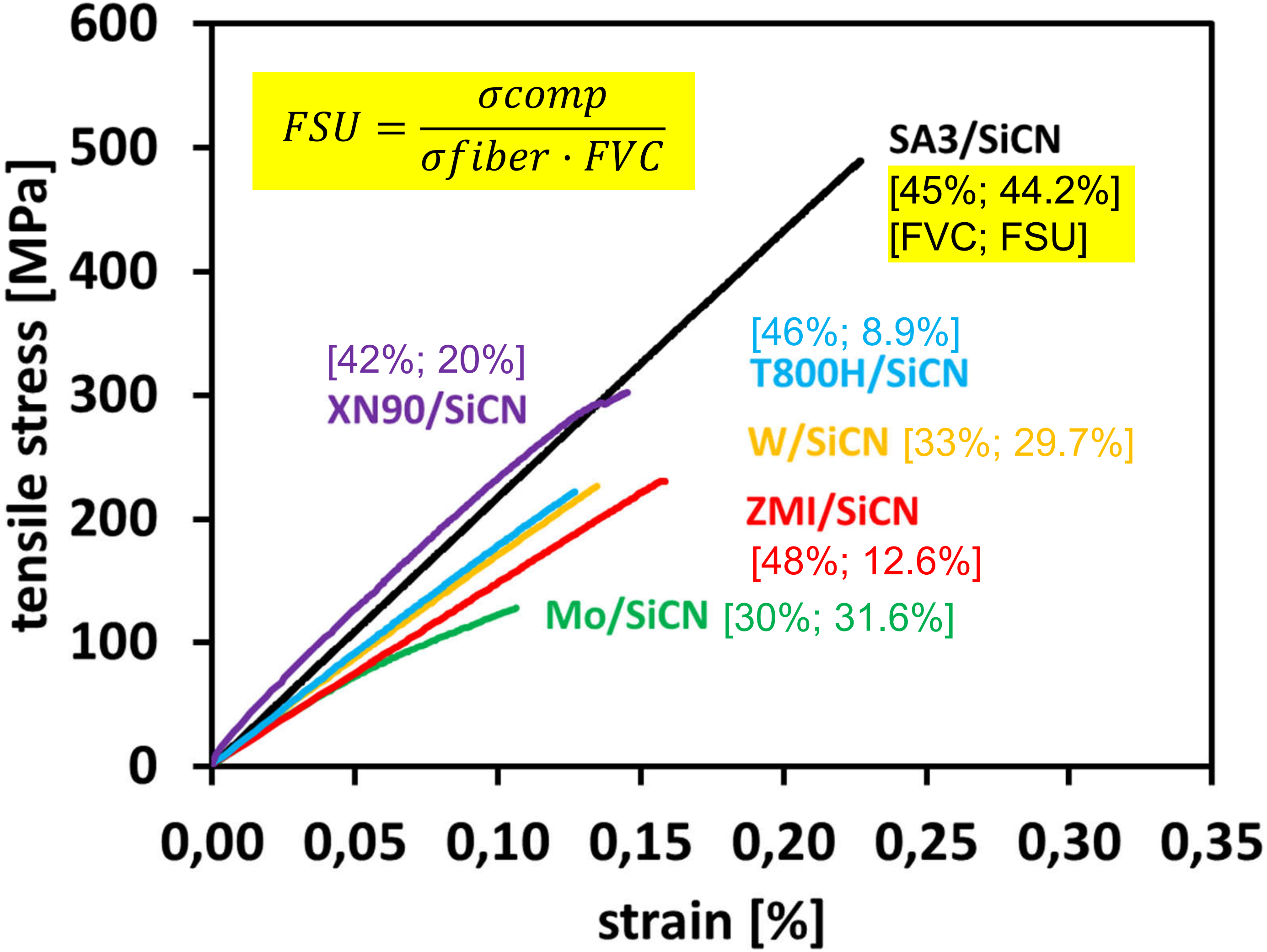
- **W + SiCN $\rightarrow$1500°C$\rightarrow$ $W + W_2C + WC + Si_3N_4$**

First mechanical models of Mo/SiCN- and W/SiCN

- Application of the model of He and Hutchinson to the new composites Mo/SiCN and W/SiCN
- Comparison to other fiber reinforced SiCN composites based on C- and SiC-fibers
- First estimations and explanations on fracture behaviour as well as damage tolerance of such composites can be foreseen

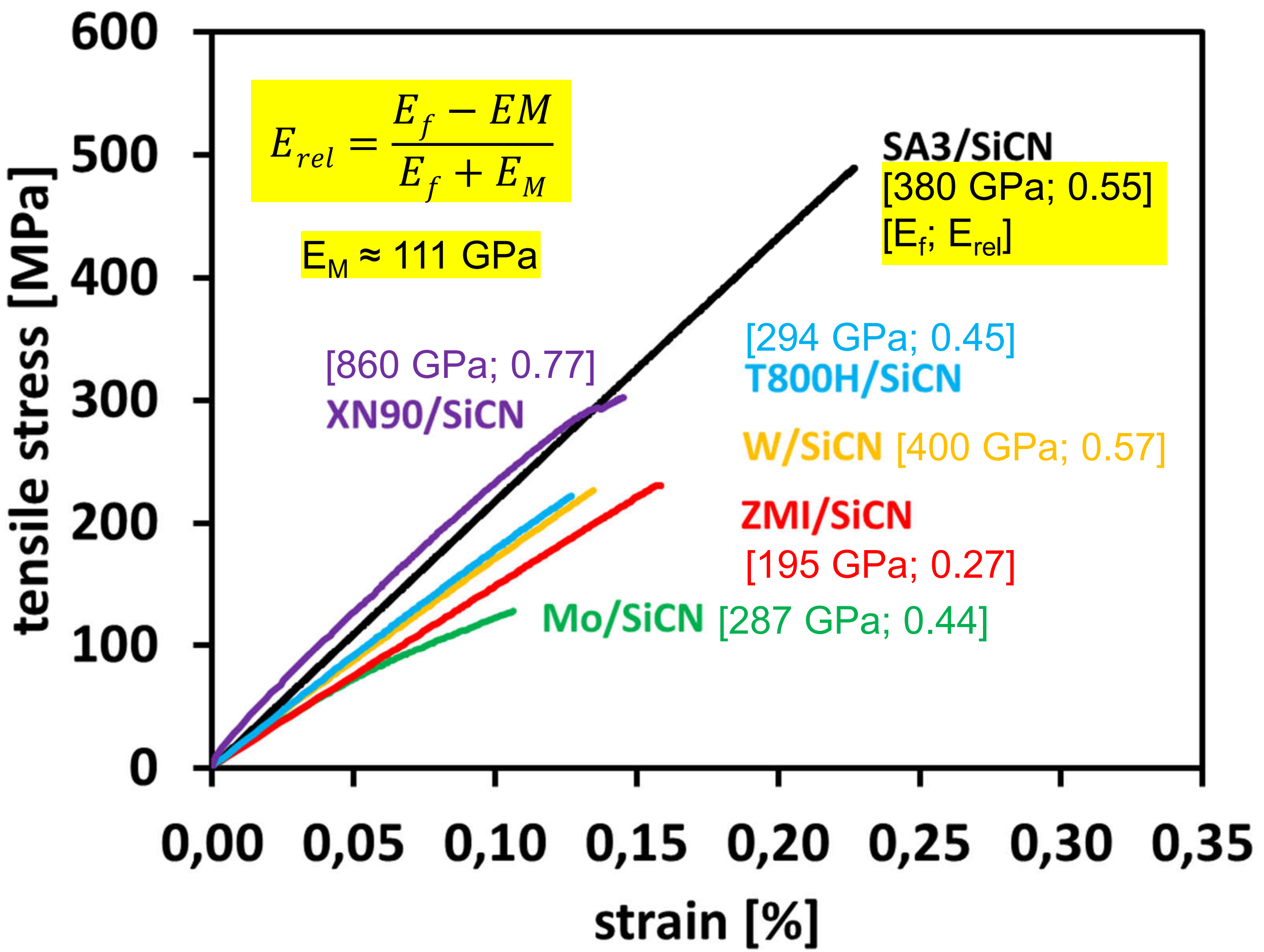
Tensile testing of various UD-fiber reinforced SiCN I

[FVC: fiber volume content; FSU: fiber strength utilisation]



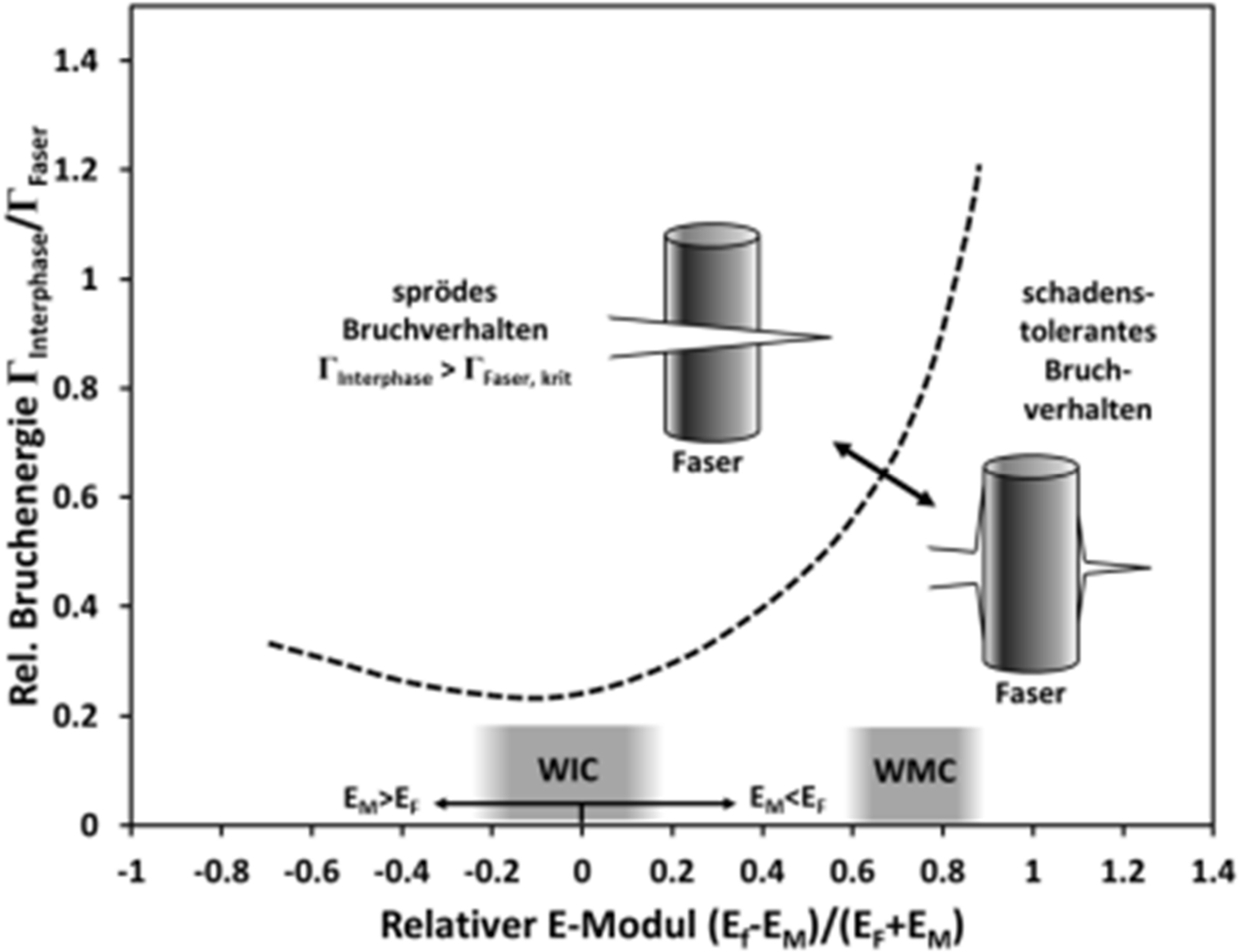
Tensile testing of various UD-fiber reinforced SiCN II

[E_f: Young‘s modulus of fiber; E_{rel}: relative Young‘s modulus of fiber and matrix]



Damage-tolerant and brittle fracture behaviour of CMCs

(Concept of He and Hutchinson)



Summary and outlook I

- Mo- and W-fiber reinforced CMCs can be easily manufactured via polymer infiltration and pyrolysis at 1300 °C (PIP)
- Mo/SiCN and W/SiCN composites are light-weight in comparison to Mo/Mo and W/W composites
- Mo/SiCN and W/SiCN show increased fracture strain compared to CMCs
- Mo/SiCN and W/SiCN can be considered as WMCs and thus need no weak interphase
- Microstructural and phase analyses have shown that Mo- and W-fibers are still present and thermally resistant in the SiCN matrix even at 1300 °C
- Thermodynamical calculations strongly recommend an additional fiber coating from C-attack!

Summary and outlook II

- Microstructural and phase analyses have shown that Mo- and W-fibers suffer from surfacial attack, mainly by C-based materials

- Applying a coating as reaction barrier (e.g. Y_2O_3) should provide further improvement in mechanical properties

- New applications are feasible due to:
    - increased fracture strain
    - good tensile and fracture strain
    - high stiffness
    - high thermal conductivity
    - low thermal expansion
    - high thermal shock resistance
    - anisotropic behaviour of composite according to tailor-made design