Metal Matrix Composite for fusion application

Background

Future energy demands cannot be satisfied with fossil fuels or renewable energy only. Furthermore, nuclear fission has significant drawbacks and risks, e.g. the radioactive waste. An alternative power source is nuclear fusion, where light atoms fuse to heavier ones in a fusion reactor by transforming mass into energy. During the fusion of the hydrogen isotopes deuterium and tritium to helium and to an energetic neutron, an energy amount of 17.6 MeV is released [1]. In magnetic confinement fusion devices, the highest loaded zone in a fusion reactor vessel is the divertor, where the fusion plasma touches the vessel wall. The divertor is exposed to heat loads of up to 20 MW/m^2 [2], [3]. Therefore the plasma facing material (PFM) in the divertor region needs an effective heat sink with cooling channels to remove the heat coming from the fusion plasma. In general, up to now the divertor is composed of two parts: the direct plasma facing material consisting of carbon or tungsten components, and the heat sink consisting of copper-rich CuCrZr alloy materials with cooling channels [4] , [5]. Future fusion reactors like DEMO will operate with coolant temperatures of at least 300°C for efficient energy production. Two different design concept are proposed: Flat-tile and Monoblock concept (Fig. 1). The heat flux loading will lead to temperatures of up to 550°C at the interface between PFM (W, C) and heat sink material (CuCrZr) [6]. The temperature gradient and the mismatch of thermal expansion of both parts of the divertor create high stresses at the interface between plasma facing material and heat sink [7]. To strengthen this zone two metal matrix composites concepts are suggested: SiC fibre [8] or W fibre reinforced copper. The fibres will contribute the strength of the composite and the copper provides the required thermal conductivity of at least 200 W/mK.

Beside the application of MMC in fusion reactors, composite materials are successfully used as heat sink materials in space applications or microelectronics. The properties of composite materials can be tailored in a wide range, mainly via an appropriate design of the interface between the fibres and the matrix, which plays a key role for the macroscopic properties [9], [10]. Thus, the main purpose of the work at the IPP MMC research group is the development of Cu/SiC and Cu/W composite materials with improved interface properties. The low interfacial shear strength between SiC fibre and Cu matrix without appropriate interface indicates a very weak bonding between fibres and matrix. For adjusting a suitable bonding strength between the composite partners we incorporated a titanium interlayer between SiC fibre and matrix as coupling agent in case of the Cu/SiC composite, followed by an adequate thermal treatment to affect chemical reactions between the titanium and the carbon coating of the fibres. To optimise the bonding strength between fibre and matrix of Cu/W composite, three interfacial concepts were compared: no interlayer between W fibre and Cu matrix; a thin PVD Cu layer incorporated between W fibre and Cu matrix; and a graded transition between W fibre and Cu matrix. By increasing gradually the concentration from the tungsten reinforcement to the copper matrix, the CTE mismatch between W fibre and Cu matrix can be reduced. The mechanical characterisation of the composite interfaces was performed using fibre push-out and pull-out tests.

Materials and Synthesis

For the long-fibre reinforcement of the copper matrix, SiC fibres (SCS 6, Specialty Materials) and W fibres (OSRAM) were used (Fig. 2). SCS 6 SiC fibres are multi-layered fibres with a diameter of 140 µm consisting of six different structural components: a carbon monofilament, thick fine-grained SiC and coarse-grained SiC-layers, a 0.5 µm thin healing layer, consisting of pure amorphous carbon, to heal surface defects and thus to improve the fibre strength and two layers of SiC doped carbon (overall thickness of outer layer ≈ 3 µm) to protect the fibre during handling and to improve the wetting properties [11]. An additional titanium layer should lead to an improvement of the bond strength between fibre and matrix, due to its reaction with the outer carbon containing layer of the fibre to TiC at temperatures above 350°C [12]. These titanium bond layers with thicknesses of 100 nm were deposited on the fibres by magnetron sputtering. An additional 500nm Cu layer was deposited to protect the Ti against oxidation.
The initial W fibres with a diameter of 100 µm were coated by magnetron sputtering with a 500 nm Cu layer or with a graded transition of 500 nm. The copper concentration is thereby gradually increased from the tungsten fibre to the copper matrix.

Subsequently to the magnetron deposition the fibres of both types were electroplated with a thick copper layer as matrix material in a $CuSO_4$ bath at room temperature. The thickness of the copper layer defines the fibre volume content in the composite: for a fibre volume fraction of 20%, the SiC fibres were coated with an 80 µm thick copper layer within 8 h. For pull-out measurements, the coated W fibres were electroplated 24 h to obtain an adequate matrix thickness (matrix thickness ≈ 1mm) to pull against. After deposition, the fibres were heat-treated at 550°C for 2 h with a very slow heating rate of 0.5 K/min to avoid the formation of pores by outgassing of hydrogen and oxygen which both are normally contained in a galvanic layer. The pores would be a result of a chemical reaction between hydrogen and oxygen to water during fast heating, which would damage the microstructure and decrease the mechanical strength ("pickle brittleness"). In the last step, coated and heat-treated single fibres were packed in a copper capsule (diameter 10 mm, length 45 mm) as dense as possible and subsequently hot-isostatically pressed at 650°C with a pressure of 100 MPa for 30 min to form the composite material. The reinforced zone had a diameter of 3.5 mm.

For the mechanical characterisation of the interface between the fibre and matrix, push-out tests on the SiC/Cu composite material and pull-out tests on coated W single fibres were performed (Fig. 3). For the push-out test the composite sample thickness and thereby the fibre length varied between 0.4 and 3 mm. Also for the pull-out test the length of the electroplated Cu matrix was varied to obtain embedded fibres with various lengths between 0.5 and 4 mm. A universal test machine was prepared to push or pull single fibres out of the matrix. Both the displacement and the resulting load have been acquired continuously. A typical load vs. displacement curve is shown in Fig. 4, which results from the push-out as well as from the pull-out tests. The curve shows an elastic increase of the load until the first local maximum is indicating the beginning of debonding. This point is called debonding load - P_d. In the further curve progression the debonding is superposed with friction. At the absolute maximum load - P_{max}, the fibre debonds completely. After debonding the force, required to overcome the push/pull-out friction decreases during the final phase of the experiment. For the push-out as well as for the pull-out tests, the P_d values were acquired as function of embedded length and serve as data which will be fitted with formulas [13] to obtain one characteristic interfacial property, the interfacial shear strength τ_d which is defined as the maximum shear stress encountered at the fibre/matrix interface just prior to the onset of debonding.

Interfacial analysis of SiC/Cu and W/Cu

The mechanical interface properties can be described by the interfacial shear strength τ_d and can be calculated from single fibre push-out or pull-out tests. The interfacial shear strength for SiC/Cu system was calculated to be 6 MPa for samples without titanium carbide interlayer and 70 MPa for samples with titanium carbide interlayer [14], [15]. As presented in Fig.5a), no reaction of the outer carbon-rich layer of the SiC fibre with the copper matrix has been observed. This layer rather acts like a lubricant, so that the fibres could be easily pushed-out by frictionless slide. The SEM image of such a slipped fibre, shows no matrix material at the fibre surface and no deformation of the copper matrix. The composites with a titanium carbide interlayer as a coupling agent exhibit complete different features: the values for interfacial shear strength and interfacial friction stress are at least 10-fold higher compared to the samples without a titanium carbide interlayer, indicating a high bonding strength between SiC fibre and copper matrix. Thus, titanium acted both chemically and mechanically to improve the bonding process. The SEM image of Fig. 5b) shows a pushed-out fibre of a composite specimen with a TiC interlayer: the carbon-rich double fibre coating of 3 µm in thickness is visible in the centre of the picture. In front of this, the titanium carbide layer and the copper matrix can be recognized. The carbon-rich double layer now remains attached to the matrix after the push-out test, in contrast to the case without TiC layer. Thus, via the TiC layer, the copper matrix is perfectly bonded to the whole SCS6-SiC fibre (comprising all their constituents), and the fibre core region itself moves longitudinally during the push-out tests by shear and displacement processes which enable a controlled energy-dissipative behaviour. The debonding processes are then governed by the thin healing layer of pure amorphous carbon between the SiC core and the outer carbon-rich double layer.

The interfacial shear strength for W/Cu system was calculated to be 26 MPa for samples without interlayer, 69 MPa for samples with a thin PVD deposited Cu interlayer and 21 MPa for samples with graded transition between W fibre and Cu matrix. Interdiffusion and segregation of W and Cu can be excluded, thus, mechanically bonding is governed. The SEM image (Fig 6a)) indicates electroplated Cu matrix residues in the coarse grooves of W fibre surface after the pull out test of the W/Cu sample without interlayer. The determined interfacial shear strength value indicates a poor mechanical interlocking. Fig 6b), showing the pulled fibre surface of the W/Cu system with Cu interlayer, indicates large amounts of Cu matrix residues on the W fibre surface which indicates a strong bonding between fibre and PVD Cu layer and which correlates to the highest interfacial shear strength. By contrast, the SEM image of the slipped fibre of the graded transition interfacial system (Fig. 6c)) shows no Cu matrix or residues of the deposited transition layer at the fibre surface which indicate the failure at the interface of W fibre and the graded transition. The value for interfacial shear strength is much smaller compared to the samples with a thin Cu interlayer, which indicates a low bonding strength for the interfacial concept with graded transition.

Conclusion

For SiC/Cu heat sink composites a titanium deposition on the SiC fibre surface with a thickness of 100–200 nm forms TiC with the carbon-rich surface layer. This leads to an improved bonding between the SiC fibre and the copper matrix. The TiC carbide formation at the fibre/matrix interface increases the interfacial shear strength by one order of magnitude in relation to composites without TiC interlayer. The shear and displacement processes at the fibre surface are then governed by the thin healing layer of pure amorphous carbon between the SiC core and the outer carbon-rich double layer of the SCS6-SiC fibre.

For W/Cu composites three interfacial concepts were analysed to develop a composite material with improved interface properties. Due to the fine microstructure of the thin PVD Cu layer incorporated between W fibre and Cu matrix and the resulting mechanical interlocking, the interfacial shear strength is increased compared to the W/Cu systems without interlayer or with a graded transition between W fibre and Cu matrix.

Acknowledgement

The authors would like to thank EADS, Ottobrunn, for performing the hot-isostatic pressing.

A part of this work has been performed within the framework of the Integrated European Project „ExtreMat“ (contract NMP-CT-2004-500253) with financial support by the European Community. It only reflects the view of the authors and the European Community is not liable for any use of the information contained therein.

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Figure captions

Fig. 1: Future demonstration fusion reactor Demo with potential divertor design concepts: Flate-tile and Monoblock

Fig. 2: Synthesis of SiC- and W-fibre reinforced copper matrix composite

Fig. 3: Schematic demonstration of a) push-out test and b) pull-out test

Fig. 4: Schematic load vs. displacement diagram for push-out and pull-out test

Fig. 5: SEM images of SiC/Cu with its failure zone: a) pushed fibre of a composite without titanium interlayer b) pushed fibre of a composite with titanium carbide interlayer

Fig. 6: SEM images of W/Cu: a) pulled fibre without interlayer b) pulled fibre with Cu interlayer c) pulled fibre with graded transition interlayer

Figures

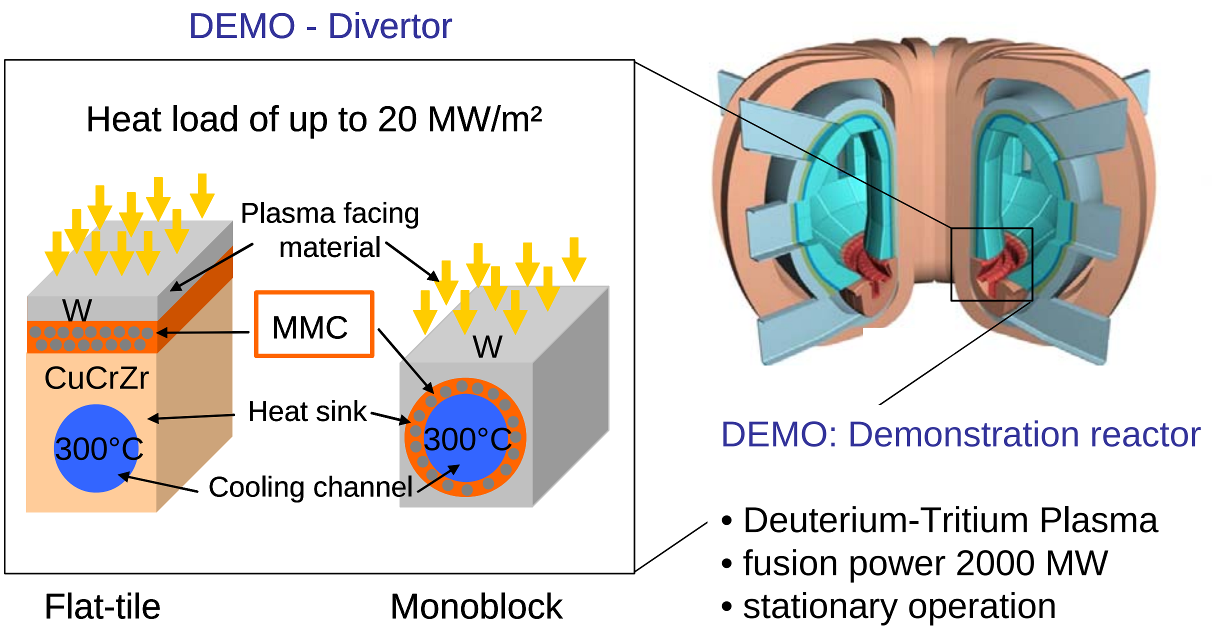


Fig. 1

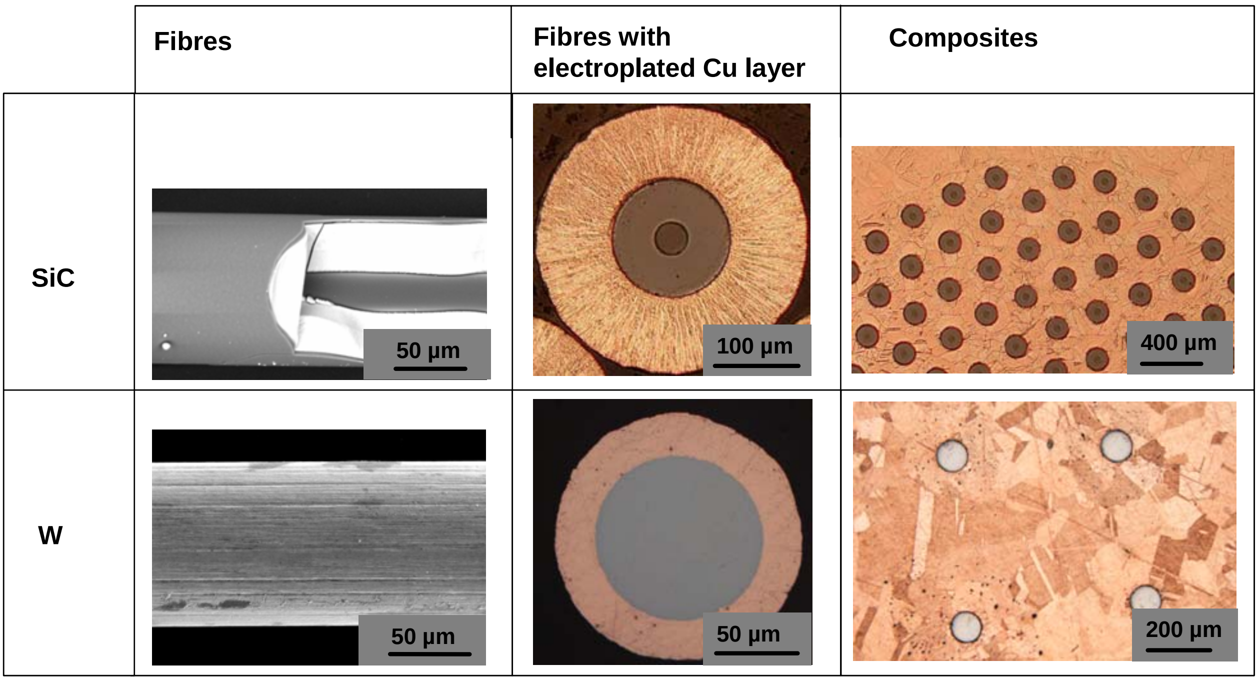

Fibres
Fibres with electroplated Cu layer
Composites
SiC
50 µm
100 µm
400 µm
W
50 µm
50 µm
200 µm

Fig. 2:

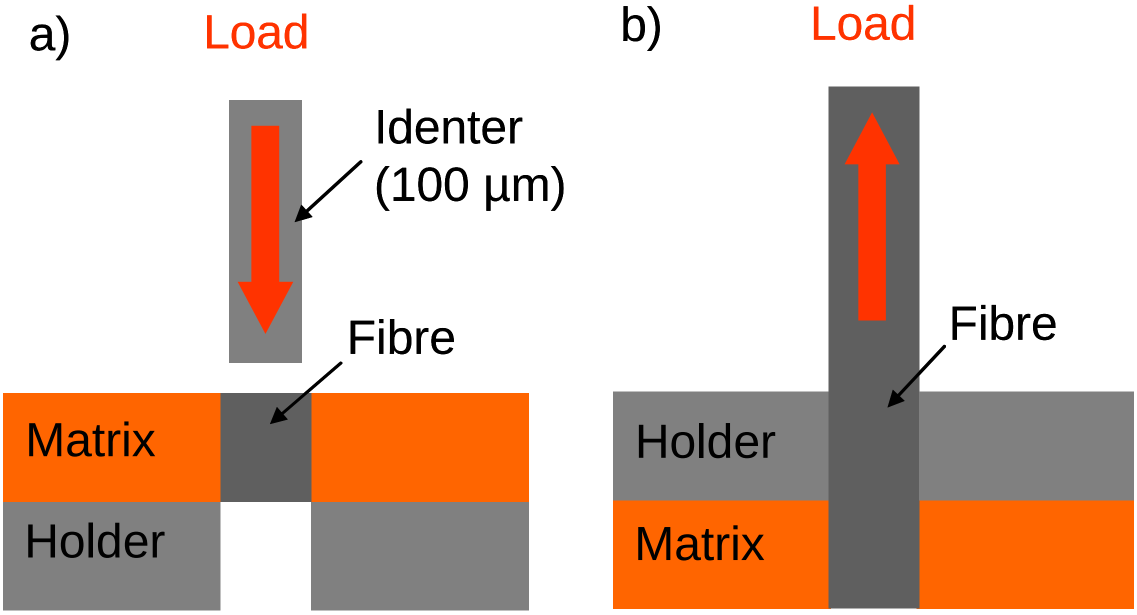
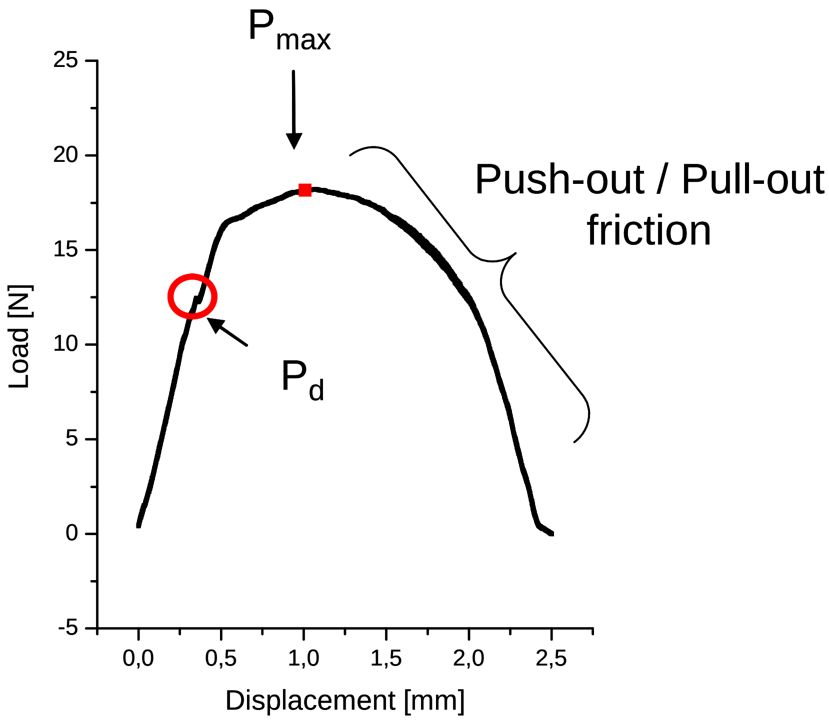

a)
Load
Identer
(100 µm)
Fibre
Matrix
Holder
b)
Load
Fibre
Holder
Matrix

Fig. 3:



P
max
25
20
15
10
5
0
-5
Load [N]
0,0
0,5
1,0
1,5
2,0
2,5
Displacement [mm]
P
d
Push-out / Pull-out
friction

Fig. 4:

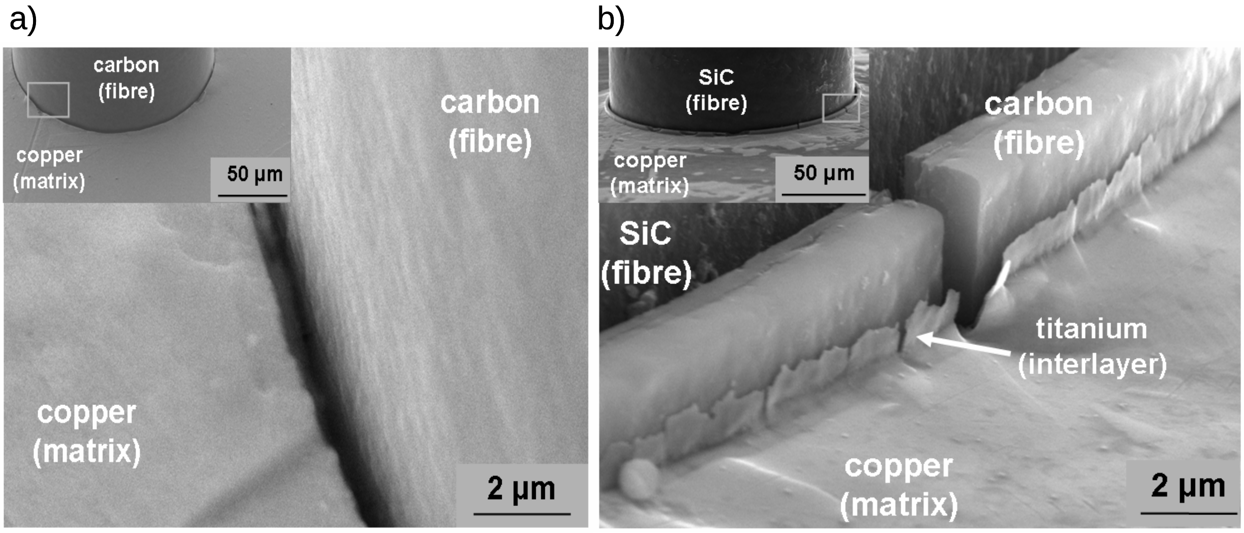


Fig. 5a) and Fig. 5b):

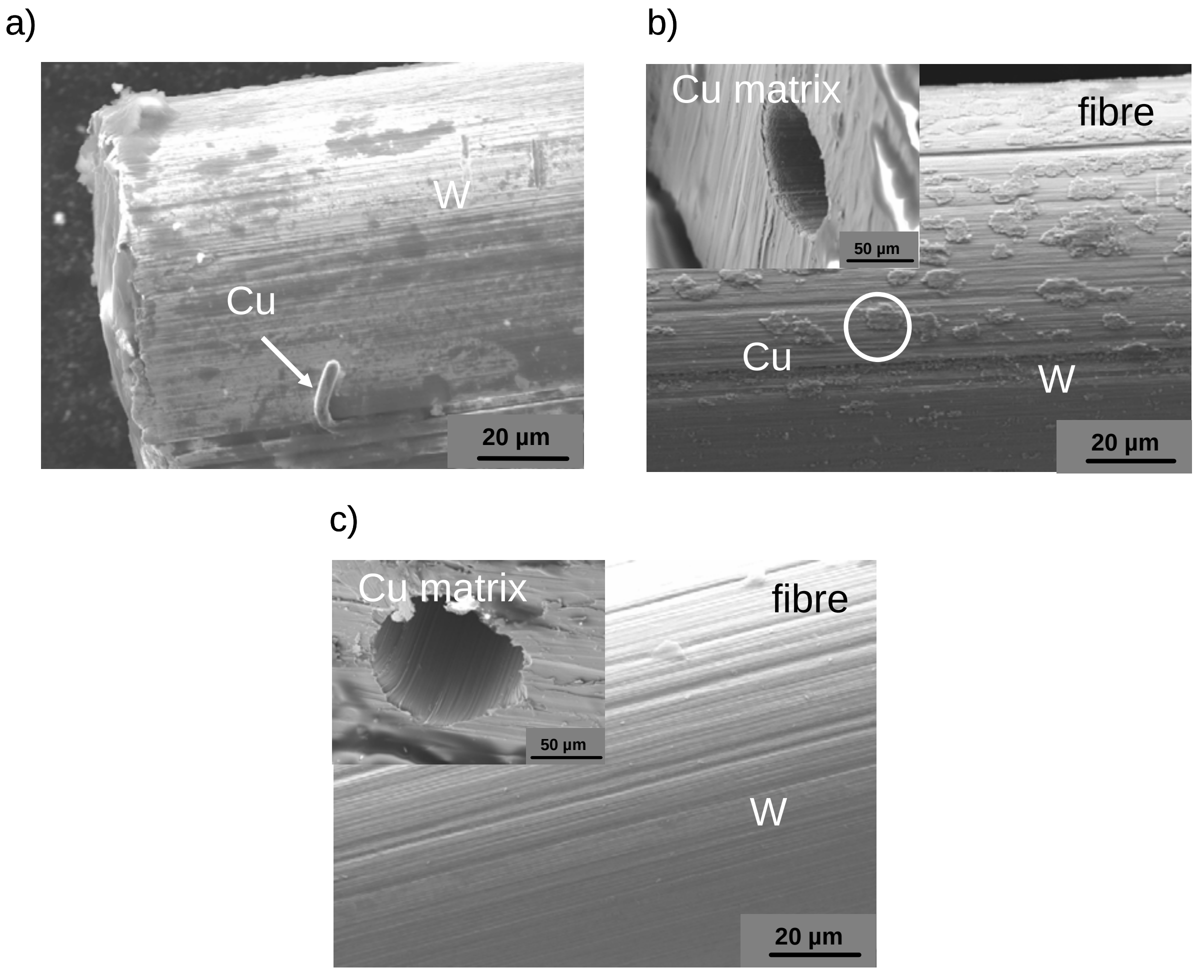


Fig. 6a), Fig. 6b) and Fig. 6c):