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European Electron Microscopy**

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**Deliverable 6.2**

**Report on protocols and standards developed in ESTEEM2**

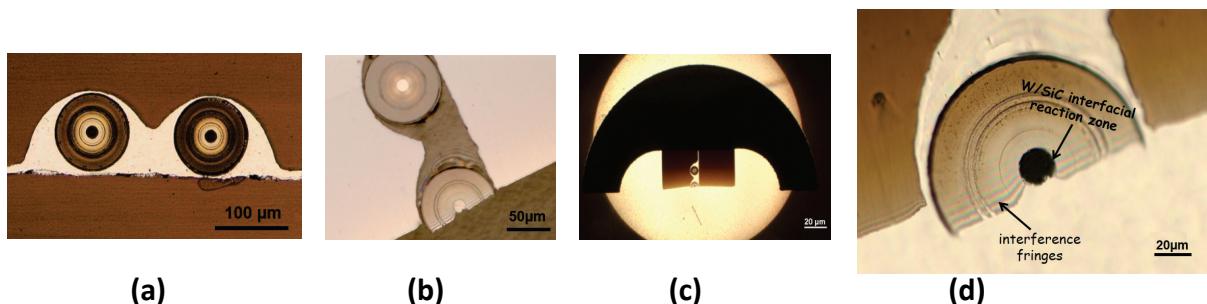
**Deliverable leader 6 – Max Planck Institute for Intelligent Systems, Stuttgart**

**Protocol contributed by the Jožef Stefan Institute, Ljubljana**

**TEM sample preparation of W/SiC composite fibers**

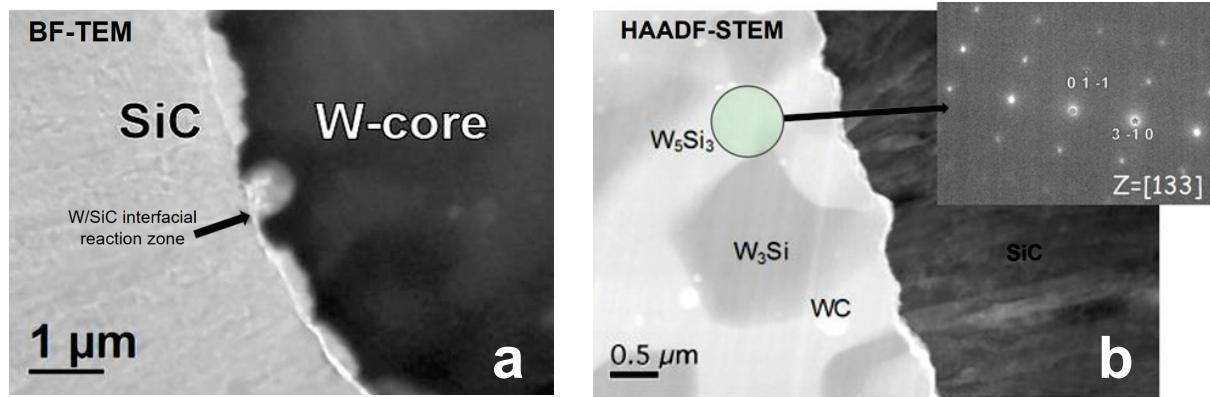
## TEM sample preparation of W/SiC composite fibers

W-core/SiC fibers TEM specimens were prepared by tripod polishing method using an automatic tripod polisher *Allied Multiprep System*. The mechanical thinning by tripod polishing of the W/SiC fibers is performed on diamond lapping films. The W/SiC specimens were embedded into epoxy resin and placed in pre-formed channels between two monocrystalline silicon plates and mounted in such a way in order to obtain cross-sections geometry of the fibers (Fig. 1a). The specimen is first polished on diamond-lapping film (DLFs) with 15- $\mu\text{m}$  grain-size, and later with 6-, 3-, 1-, 0.5-, and 0.1- $\mu\text{m}$  grain DLFs. The final polishing step is done on a polyurethane cloth using a silica solution with 50-nm particle size (*Allied Colloidal Silica Suspension*). During every polishing step, the specimen is regularly monitored under the OM. Prior to polishing the other side, the sample is removed from the pyrex by heating on a hot plate. In order to polish the second side, the specimen is turned upside down and glued onto the pyrex specimen holder again. The specimen is polished down to a thickness of app. 180  $\mu\text{m}$  using a 9- $\mu\text{m}$  DLF, where a wedge angle of 1.5° is introduced. The sample is then polished down to 70, 50, 30, 10  $\mu\text{m}$  using 15-, 6-, 3-, 1-  $\mu\text{m}$  DLFs, respectively. At 10- $\mu\text{m}$  thickness the load is removed and the specimens are additionally polished with a 1- $\mu\text{m}$  DLF and Green Lube until interference fringes are visible (Fig. 2b). A final polishing step is performed with 0.5- $\mu\text{m}$  DLF and 50-nm colloidal silica to further thin the specimen wedge to electron transparency. After finishing all the polishing steps, the sample is glued to 3 mm Cu support half-grid (Fig. 2c). The wedge-shaped specimens are additionally cleaned and Ar<sup>+</sup> ion-beam thinned in a Gatan PIPS at 2.0 keV for 30 minutes and at 0.6 kV for 15 min (Figure 2d).



**Figure 1:** Optical micrograph showing various steps of tripod polished specimen by wedge-shape method of W/SiC with subsequent ion-milling.

Described TEM sample preparation yields electron transparent regions of SiC, W and SiC/W interface as shown if Figure 2.



**Figure 2:** (a) TEM image of W-core/SiC interface. (b) STEM/HAADF micrographs of W/SiC interface.