

TEM specimen preparation of a SiC/SiC composite by conventional ion-milling, tripod polishing and focused ion beam (FIB): a comparative study

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Composite materials made of continuous crystalline SiC fibers within a submicron polycrystalline SiC ceramic matrix can be used for the first wall of a future fusion reactor, due to their excellent mechanical properties [1]. These SiC/SiC composites are processed at higher temperatures than SiC fibers or a combination of SiC particles and sintering additives with a low-melting-point eutectic composition based on the Al-Si-P-O system. The interface between the SiC fibers and the submicron SiC matrix largely determines the mechanical properties of the composite under a load. In order to improve these properties, the SiC fibers were coated with a thin layer of diamond-like-carbon (DLC) by physical vapor deposition (PVD). Afterwards, transmission electron microscopy (TEM) analyses were performed in order to observe the structure and chemical composition of the interface between the SiC fibers and the SiC matrix after a thermal treatment at 1300°C for 3h in an argon atmosphere. Our previous results showed that a reaction layer formed between the thin layer and the matrix. In order to apply an optimized TEM sample-preparation procedure for the SiC/SiC composite, three specimen-preparation techniques were employed: conventional ion milling, tripod polishing and focused ion beam (FIB). The results of these three preparation approaches are described and compared.

The TEM specimens prepared by the conventional method were first mechanically grinded to a thickness 100 μm, followed by dimpling in cross-section geometry down to a thickness of 16 μm. Afterwards, the specimens were Ar-ion thinned at 4 keV and 10° incident angle to perforation in a Bal-Tec RES 010 ion-miller (Fig. 1a).

For the TEM specimens prepared by the mechanical polishing method an automatic Allied MultiPrep System was used [2]. The SiC/SiC specimens embedded into an epoxy resin were mounted in such a way that cross-sections of the fibers were obtained. The specimens were mechanically polished on a diamond-lapping film (DLF) at a small wedge angle of 1°. The final polishing step was performed on both sides of the sample by using colloidal silica to thin the specimen to electron transparency. Afterwards, the specimens were glued on a Cu grid. Additionally, the specimens were Ar-ion thinned at a low energy of 1.5 keV for 10 min using a Gatan PIPS (Fig. 1b, c).

For the TEM specimen preparation using FIB (Carl Zeiss Cross Beam 1540) TEM sections of the SiC/SiC composite were prepared with the in-situ, lift-out technique [3]. A linear Pt strap was deposited on the area of interest in order to protect the specimen as well as to improve the mechanical support during the milling process. The specimens were further milled using a FIB with the Ga ions operating at 30 keV. Using the in-situ micromanipulator (Fig. 2a), the TEM lamella was lifted-out and positioned on a Cu-grid. The pores in the SiC matrix were filled with Pt (Fig. 2b). Afterwards, only the selected parts of the TEM lamella were thinned with Ga ions to a thickness of around 50–70 nm (Fig. 2c). In this way the prepared specimens were thin enough for TEM observation (Fig. 2d).

The properties of the SiC fibers and the SiC matrix are quite different, which resulted in a preferential etching of the SiC matrix for samples prepared by conventional ion-milling. In the samples prepared by wedge-shaped polishing the thickness of the SiC fiber was almost double compared to the SiC matrix. The subsequent ion-milling resulted in a porous matrix. However, the thickness of the fibers was still too thick for TEM measurements. In contrast, the samples prepared by FIB offer a uniform thickness across the SiC/SiC composite material. The important advantage of

FIB sample preparation compared to the other two above mentioned methods was the ability to strengthen the SiC matrix by filling the pores with Pt. Additionally, by thinning only the selected parts across the fiber–matrix interfaces, and not the whole specimen, the TEM lamella was robust enough to handle and thin enough for the TEM experiments. Using this approach, preferential etching of the SiC matrix compared to the SiC fibers was strongly reduced.

References

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 [4] The authors acknowledge financial support from the EU under the Framework 6 program under a contract for an Integrated Infrastructure Initiative. Reference 026019 ESTEEM. We thank Ute Salzberger (MPI, Stuttgart) for helpful discussions and technical support regarding the tripod sample preparation method.

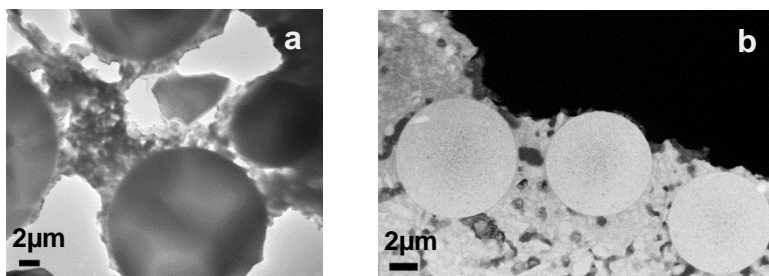


Figure 1: (a) Bright-field TEM image of ion-milled SiC/SiC composite. (b) HAADF-STEM image of the specimen prepared by wedge-shape polishing technique.

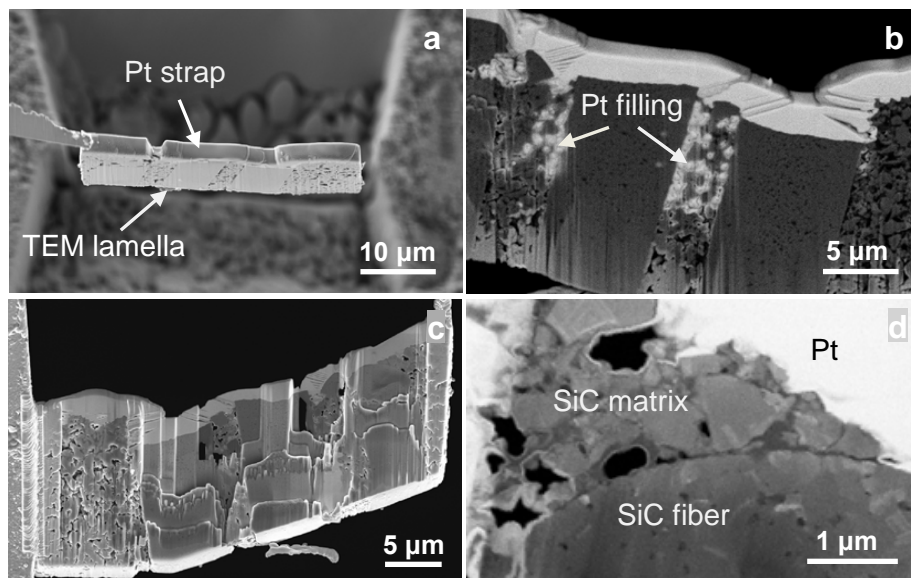


Figure 2: The FIB in-situ lift-out technique for preparation of a cross-section from a porous SiC/SiC composite material: (a) Sectioning of a thin lamella using focused Ga ions at 30 keV. The micromanipulator assisted in the extraction the lamella. (b) Filling of the pores within the SiC matrix with Pt prior to the thinning. (c) The TEM lamella after the final thinning. (d) ADF-STEM image of the cross-section of the SiC/SiC thin foils.