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PROOF-OF-CONCEPT EXPERIMENT REPORT

QUALITY EVALUATION OF THIN COATINGS ON POROUS CERAMIC SUBSTRATES

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Proof-of-Concept experiment details

Analyzed samples:

- bare porous ceramic SiC

- W coated porous ceramic SiC (sample prepared by Dr. Cristofori @UNIVE and analyzed by Prof. Fanetti @UNG)

Description by company: the sample consists of a porous ceramic based on silicon carbide with a tungsten metallic coating. The thickness of the coating is estimated by the company between 500 nm and a few microns. (The proposal initially envisaged a chromium nitride coating which was not possible to obtain from the company for technical reasons).

Sample preparation:

- Fracturing; Embedding in epoxy resin; Cutting and mechanical polishing (down to 3 µm diamond abrasive) @UNIVE

- X-section Ar-ion polishing @UNG

- 6 nm C conductive coating @UNG

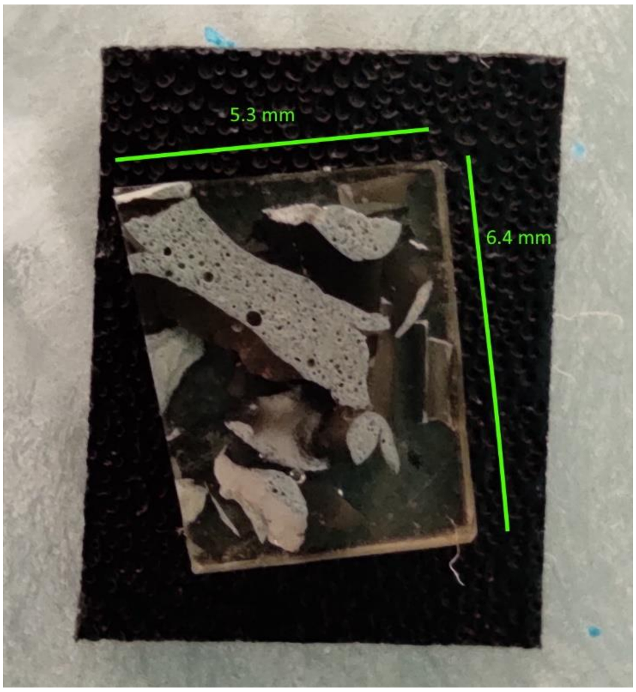

Figure 1: Picture of W-coated sample embedded in epoxy resin, after cutting and mechanical polishing.

Analysis:

SEM images in Variable Pressure (VP) chamber @UNIVE

Bare porous SiC (after fracturing, with no further sample preparation) and W-coated porous SiC (after mechanical polishing) were observed by VP SEM. Examples of SEM images are reported in Figure 2.

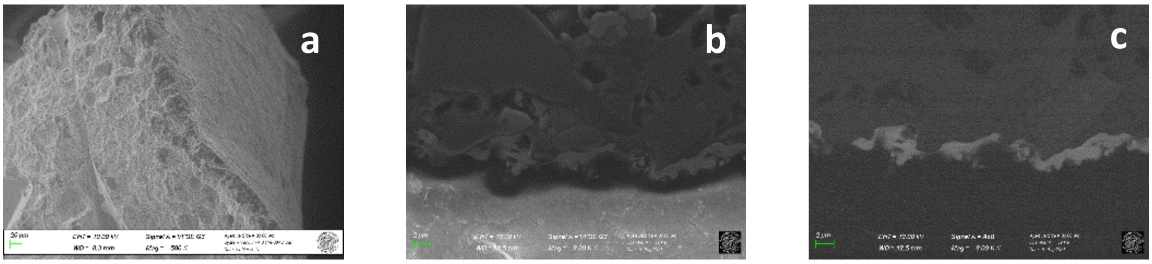

Figure 2: SEM images of bare porous SiC after fracturing (a) and W-coated SiC. after fracturing, embedding in epoxy resin, cutting and mechanical polishing (down to 3 µm diamond abrasive) observed by secondary electrons (b) and back scattered electrons (c).

SEM images in High Vacuum (HV) chamber after C conductive coating @UNG

In Figure 3, SEM images in HV chamber of the W-coated porous SiC are shown.

- <u>W-coated porous SiC</u> - after fracturing, embedding in epoxy resin, cutting and mechanical polishing (down to 3 µm diamond abrasive)

Secondary Electrons | Back Scattered Electrons

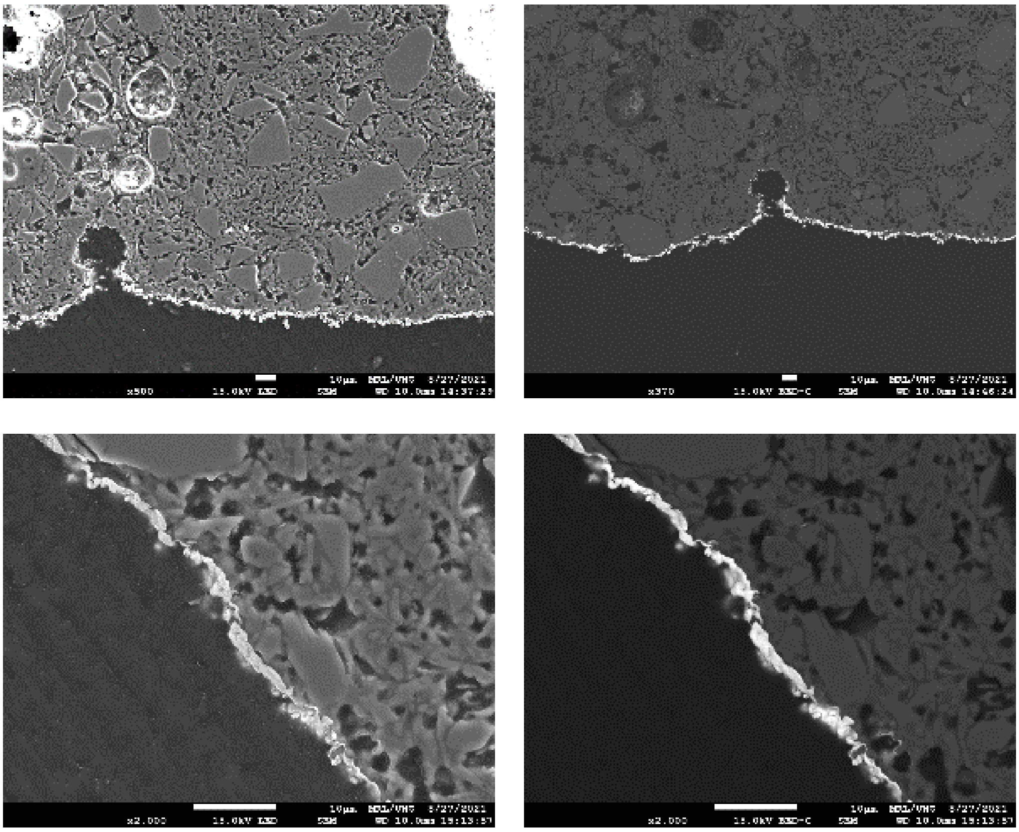

Figure 3: SEM images of W-coated SiC. after fracturing, embedding in epoxy resin, cutting and mechanical polishing (down to 3 µm diamond abrasive) observed by secondary electrons (left) and back scattered electrons (right).

The W coating is clearly visible from BSE images as brighter regions. The covering is not always homogeneous, and some areas seem not covered at all.

Two hypotheses: or the mechanical polishing removed the coating layer or some surface areas of the SiC are not reached by W during the deposition. For this reason, X-section Ar-ion polishing has been performed. SEM images are shown in Figure 4.

Secondary Electrons | Back Scattered Electrons

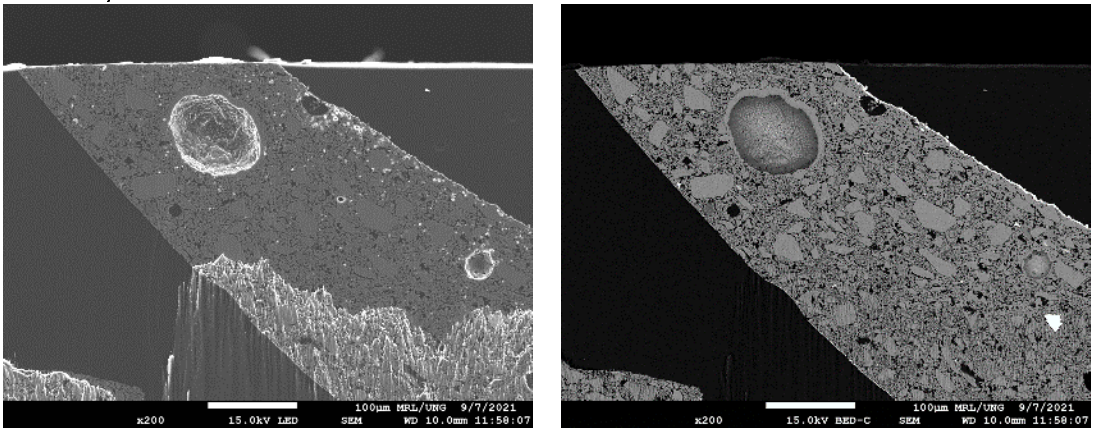

Figure 4: SEM images of W-coated SiC. after mechanical polishing and X-section Ar-ion polishing observed by secondary electrons (left) and back scattered electrons (right).

The morphology of the W coating seems like that observed previously on the mechanically polished sample.

It is confirmed that the coating is not covering homogeneously the surface of the sample.

From BSE images it is possible to estimate the thickness of the W coating (Figure 5).

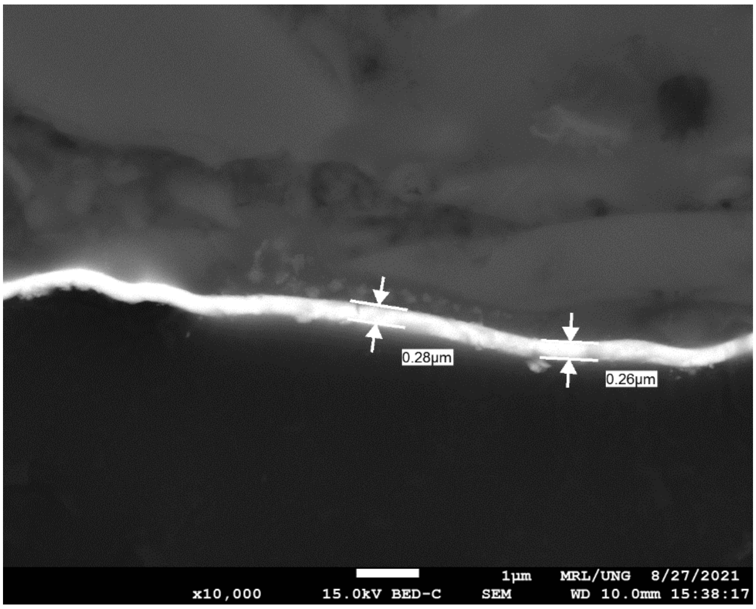

Figure 5: BSE images of W-coated SiC. The thickness of the W metallic layer in this region is (0.27±0.01) µm.

Some differences between Ar-ion polishing and mechanical polishing:

- The X-section is smoother with Ar polishing, but for this sample it doesn't seem relevant, especially if observed with backscattered. However, on the left side in certain areas a very thin layer of W is visible. Probably in this area the precursor arrived much less, but it did. Perhaps with a mechanical polishing we would not have seen this feature.

- The pores are hollow, where with mechanical polishing they usually fill with residue.

- With X-section polishing, the processed area is obviously smaller than mechanical polishing, so it can be analyzed with less statistics. On more homogeneous samples this is not a problem, but on samples where the variations are on a large scale, it can be relevant. For example, in this X-section we have 2 interfaces, one with coating and one without. Previous measurements had shown that uncovered interfaces are fewer than covered ones.

Energy Dispersive X-ray spectroscopy (EDX)

EDX data show that the metal coating is W. Al impurities are detected.

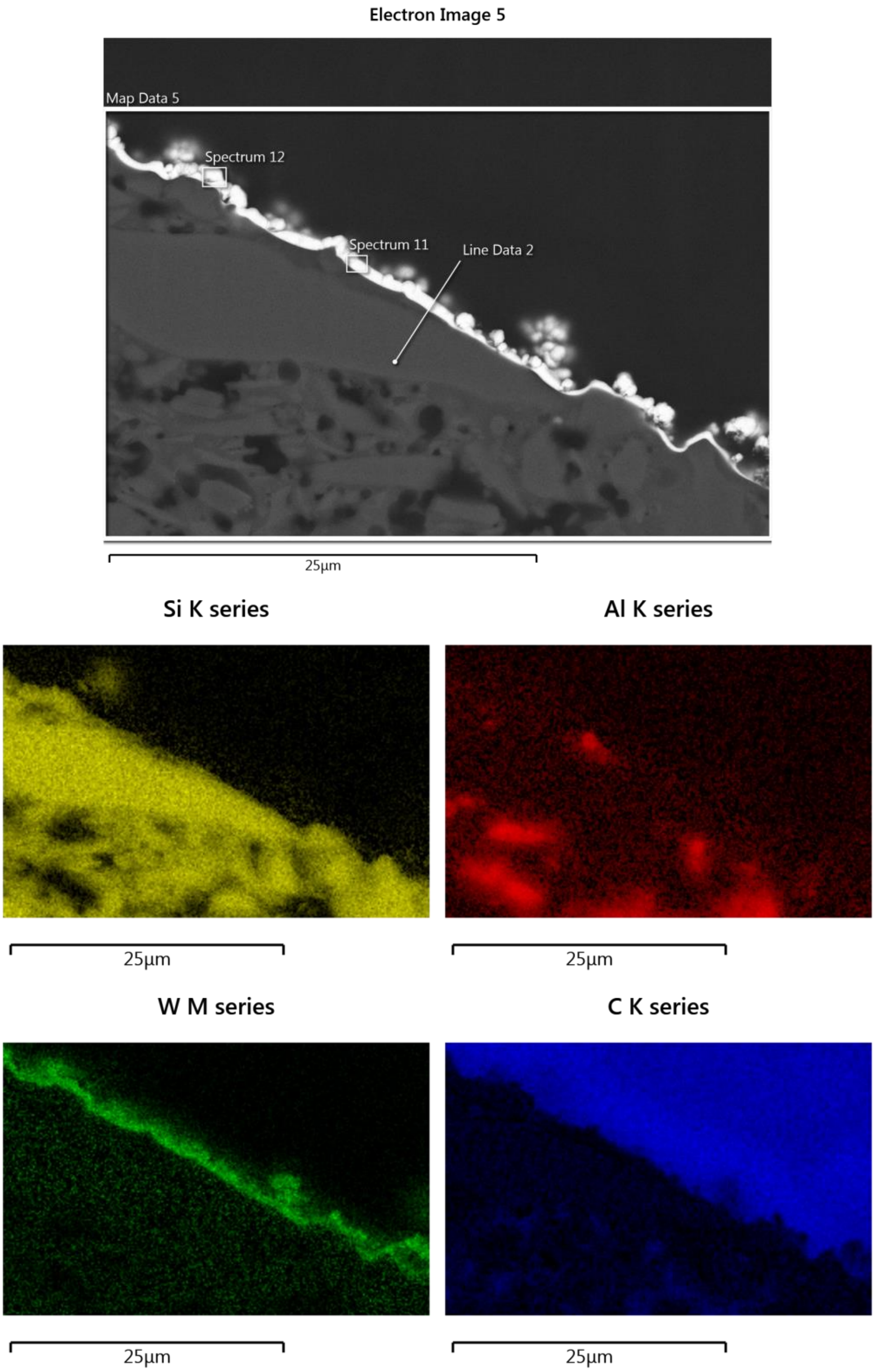

Figure 6: SEM (top) and EDX maps of Si, Al, W and C. Observation conditions: Imaging: beam energy 10-15 KeV; secondary electron detector. EDX mapping and spectroscopy: beam energy: 15 KeV.

Conclusions:

- After the successful preparation of the sample by mechanical polishing and Ar-ion polishing, it was possible to observe the sample in cross section by SEM. Mechanical polishing is effective in the preparation of this kind of samples for electron microscopy, and optimal for large area overview and statistics. On-contrary, X-sectioning by Ar ion milling can provide a locally smoother x-section surface, which allows the investigation of the morphology without artefacts and at higher resolution (e.g. the local W coating morphology).

- The Back Scattered Electrons images are effective in showing and distinguish the metallic coating, and it was possible to observe the homogeneity of the coating and estimate its thickness.

- EDX investigation confirmed the elemental composition of the coating and revealed Al impurities in the SiC matrix. EDX can be effectively used in the study of this system, to investigate the ceramic composition, find impurities and identify the metal coating.

This report has been written by Federica Rigoni on 03/06/2022