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TEM Investigations of Multi-Layer Selective Absorber thin films for concentrated solar plant: structure and composition

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Background and Method

The solar energy resource is still relatively under-exploited although it could cover most of our energy needs. Beyond the development of photovoltaics, there is a worldwide challenge to deploy large-scale concentrated solar thermal power plants. To address this issue, materials with high absorption in the visible range, low emissivity and good temperature resistance are required. To this end, multi-layered optical thin films produced by plasma processes are developed in the NANOPLAST project (nanoplast-project.cnrs.fr, ANR-19-CE08-0019). The stack encompasses an anti-reflective top-layer based on tantalum oxide or oxynitride, a selective absorber in the W-Si-C ternary system and a tungsten Infra-red reflective layer, denoted [Ta-O-N]/[W-Si-C]/[W]. These films are deposited by plasma-assisted technique: first Direct Current sputtering from a W target for the IR reflector, second dual reactive magnetron sputtering and Plasma Enhanced Chemical Vapour deposition, from a W target combined with different amounts (5% to 28%) of TMS-Si(CH3)4 diluted in Ar and finally reactive magnetron sputtering by adjusting O2 and N2 for Ta-O-N top layer[1].

Results

TEM characterisations were carried out to get insight into the structure and composition of the absorber, the anti-reflective top films and then the annealing of the complete system. Particular attention was paid to the influence of sample preparation. FIB (Focused Ion Beam) and ultramicrotomy were carried to get insight in the homogeneity along the growth direction of the multi-layer. In this work, we want to highlight the complementarity of both sample preparations for TEM. Actually if FIB is now well-established and allows to precisely select the region to be analysed, in the present case due to the non-equilibrium deposition conditions, FIB could also induce artefacts due to differential sputtering and/or re-deposition of elements (such as Ga) that may lead to misinterpretations. Hence by observing cross-sections prepared by ultramicrotomy, we were able to dismiss possible impacts of FIB on the observations thanks to the adhesion of the whole stack.

Therefore, for the absorber, depending on the tetramethylsilane (TMS-Si(CH3)4) fraction in the Ar discharge, the quantity of Si introduced in the W-SiC:H material can be tuned with the objective to explore the possibility to prepare nanocomposite thin film composed of W nanoparticles dispersed in a dielectric SiC:H matrix. In dedicated conditions (5 and 8% TMS), FIB preparation and ultramicrotomy confirmed that W nano-crystallites incorporating C atoms could actually be identified by coupling STEM-HAADF (Figure 1.a) (Scanning Transmission Electron Microscopy - High-Angle Annular Dark Field) images, EDS mapping (Figure 1.b) and High Resolution imaging. In other conditions (20 and 28% TMS), amorphous layers are observed.

Next, the annealing of the layers was studied, first the absorber alone and then on the complete multilayer device (Figure 1.c multilayer before annealing). We thus highlighted the influence of the annealing mode (air or vacuum) and its duration on the structure, composition and therefore properties of the thin film.

Conclusion

These STEM analyses provided crucial information on the structure of the active layer and on the evolution of all layers after thermal annealing. Such information is crucial for optimizing the deposition process, especially concerning its intended application. In addition, this study illustrates the complementarity between sample preparation by FIB and ultramicrotomy. Selecting regions of interest for the FIB preparations can bring more interesting results compared to random slicing by ultramicrotomy whereas the latter allows ascertaining results observed on FIB cross-section thanks to its less damaging conditions.

Graphic:

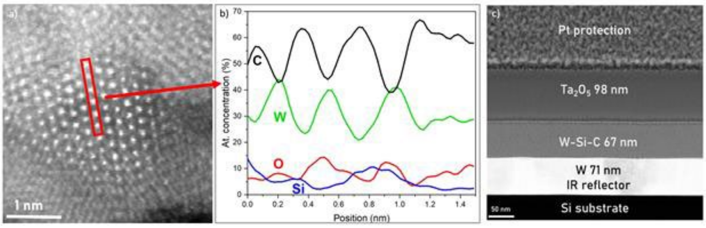

Figure 1 : a) STEM-HAADF images of nano-crystalites (ultramicrotomy preparation) and b) its corresponding EDX profiles; c) STEM-HAADF image of the complete stack before annealing (FIB preparation)

Keywords:

STEM-HAADF, Multilayer coating, FIB, Ultramicrotomy

Reference:

[1] A. Diop et al., ‘Comprehensive study of WSiC:H coatings synthesized by microwave-assisted RF reactive sputtering’, Surf. Coat. Technol., vol. 459, p. 129408, Apr. 2023