	Name of the PhD candidate: **Johann COLAS** Title of the PhD thesis: **Study of the behavior of multilayered multifunctional coatings at high temperature** Dates (start/end): 01/10/2016 - 30/09/2019
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Other contributor	Co-funding ANR and SOLSTICE

Context and objectives

Solar Tower Power Plants (STPPs) can be improved by the use of Brayton's thermodynamic cycle with gas as heat transfer fluid. The gas temperature at the turbine inlet must be higher than 1300 K. The materials that are currently used for the solar receivers do not afford them to reach this temperature. Moreover, at high temperature, the material must possess a high creep resistance, a low oxygen reactivity, an elevated thermal conductivity and a high solar absorptivity. No bulk material can meet all these constraints, therefore the selected solution consists of ceramic coating over a metallic substrate. The low oxygen reactivity is ensured by the ceramic coating and the substrate guarantees good mechanical strength and thermal conductivity. The aim of this PhD is to find materials that could be used as substrate and as coating above 1300 K and then to characterize the oxygen reactivity and the impact of the oxidation on the radiative properties of the substrate/coating(s) composite (SCC).

Approach

This PhD thesis is divided in three tasks:

- Selection of some accurate couples of materials (coating and substrate) based upon a bibliographic review
- Experimental studies on the selected substrate/coating(s) composites. The samples are produced at SIMaP-CNRS, Grenoble, France using High Temperature Chemical Vapour Deposition (HTCVD) and oxidized using the REHPTS (high pressure and solar temperature reactor, in French *Réacteur Hautes Pression et Température Solaire*) at temperatures from 1400 to 1800 K
- Characterization of the oxidized samples to determine how the structural and thermo-radiative properties would be affected by the oxidation

Main results

Two coatings can be deposited using HTCVD on various metallic substrates: aluminum nitride (AlN) and silicon carbide (SiC), the deposition of SiC requiring the pre-deposition of an AlN accommodation layer. These materials possess an excellent oxidation resistance but cannot be used as substrate because of their brittle behavior.

The literature review has permitted to select 3 kinds of candidates for the substrate:

- The Mo-based refractory alloy TZM possess very good mechanical and thermal properties, and a coefficient of thermal expansion (CTE) close to the one of AlN but their oxidation resistance is very poor due to the production of gaseous molybdenum oxide
- Fe-Cr-Al alloys (as FeCrAlloy or Kanthal APMT) have a good oxidation resistance, but their melting point is relatively low (< 1800 K). Moreover, their CTE are superior to the one of AlN, which may lead to a cracking and a spalling of the coating during heat treatments
- Mo-Si-Al alloys (as Kanthal Super ER - KSER) have an excellent oxidation resistance, and a CTE lower than the one of the Fe-Cr-Al alloys and slightly higher to the one of AlN. It could be an alternative material that will not oxidize as drastically as the TZM alloy, and will less expand than the Fe-Cr-Al alloys.

TZM was coated with AlN and FeCrAlloy and Kanthal APMT was coated with AlN or AlN/SiC bilayer. Coated TZM was quickly oxidized due to the presence of porosities, although the coating does not crack or spall during the high temperature experiments. Fe-Cr-Al alloys coated with AlN or AlN/SiC seem to possess a higher oxidation resistance than the as received sample [Fig. 1 a].although cracks appear due to thermal cycling [Fig. 1 b]. Moreover, the coatings improve the room temperature solar absorptivity. Preliminary results on the KSER alloy demonstrated that this material could support a cyclic oxidation with reduced damages up to 1800 K.

Remaining issues, proposed solutions and future works

The sealing of the coating on TZM is an important issue to prevent it from a severe oxidation, and SIMaP laboratory managed to improve this sealing using CVD to enrich the Si content of the TZM at the interface with the coating. No porosities were therefore found through the coating and the coated materials were able to stand inside a resistive furnace more than 96 h in air at 1400 K without damages on the substrate.

Cracks on coated Fe-Cr-Al alloys are due to the CTE mismatch and can be avoided by a bond coat or by using a substrate with a lower thermal expansion such as KSER. High temperature emissivity measurements are planned for Fe-Cr-Al alloys native, pre-oxidized and coated with AlN and SiC/AlN. The deposition of AlN and SiC/AlN coatings on KSER substrates is under investigation at SIMaP.

a) *b)*

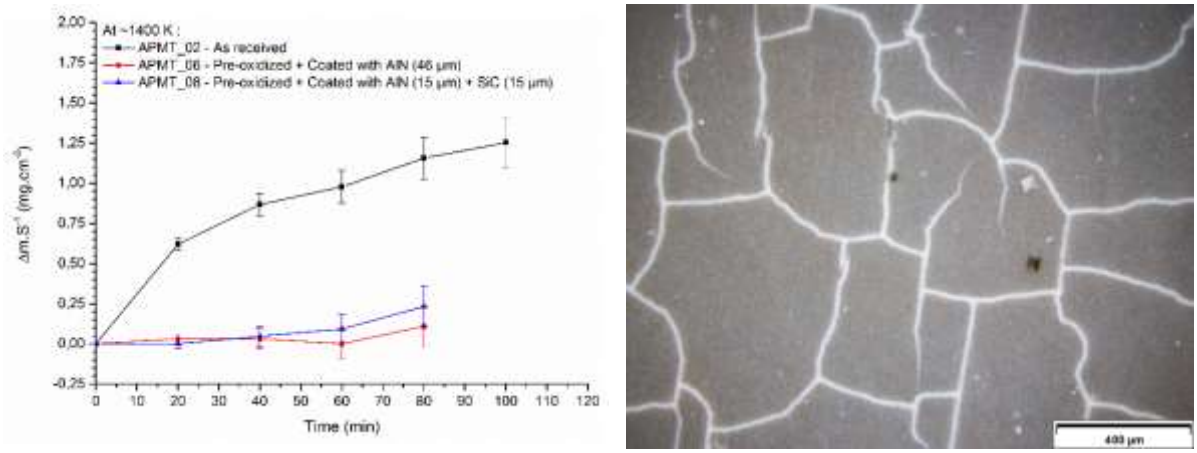

Figure 1. Evolution of the mass change after several cycles of 20 min. oxidation in air at 1400 K (a), and optical picture of AlN/SiC coated Kanthal APMT after 3 cycles of 20 min. oxidation in air at 1400 K (b)

International Conferences

- J. Colas, L. Charpentier, M. Balat-Pichelin, M. Pons, F. Mercier, D. Chen, D. Pique, “Materials for very high temperature solar receivers”, *14th International Ceramics Congress (CIMTEC),* Perugia (Italy), June 4-8, 2018