AP-CVD ZrB_2 PROCESS DEVELOPMENT FOR DISCRETE AND DUPLEX UHTC COATINGS

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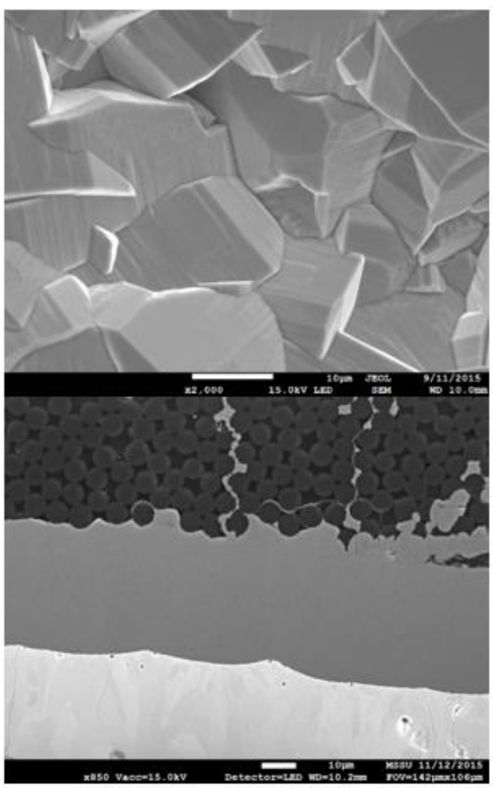
Refractory borides are a class materials with properties ideally suited to extreme operating conditions such as leading edges, spaceplanes, hypersonic vehicles and volumetric solar power receivers. The ability to coat complex geometries and varying materials with refractory borides such as zirconium diboride, (ZrB2), is offered through chemical vapour deposition, (CVD), methods. CVD ZrB_2 can offer high purity, uniform and fully dense coatings economically, negating many of the current issues with powder processing methods that dominate commercial availability.

The study was conducted to establish ZrB_2 coating processes by atmospheric pressure CVD, (AP-CVD), on a variety of substrate materials including graphite, CMCs, SiC coated CMCs, and C and SiC fibre fabric. Single layer coatings were developed, although these are limited in temperature performance, as well as multilayer ZrB_2-SiC coatings to offer an innovative and effective solution to operation in the high and ultra-high temperature regimes. Literature studies demonstrated CVD processes could be achieved through direct chlorination of zirconium metal to generate a metal chloride precursor that underwent subsequent reduction with BCl_3 and H_2 to generate ZrB_2 coatings. An iterative process of parameter selection and modification, deposition trials and analysis was undertaken to evaluate optimal deposition conditions.



Uniform, cohesive and conformal coatings were obtained with minimal contaminants and an approximate 2:1 stoichiometry of B:Zr through EDX analysis. Coatings were high purity, highly dense and demonstrated favourable adhesion between the ZrB_2 and SiC layers, (See Figure 1 below for ZrB_2 surface and multilayer cross section) Fibre coating showed particulate formation on the individual fibres but not complete coalescence of the coating. The ZrB_2 formed distributed crystals on the fibres rather than a continuous uniform coating. There is also a strong gradient from the outside of the fibre bundle to the inside.

The study was successful in developing a small-scale plant capable of depositing high purity, stoichiometric ZrB_2 coatings through an optimized CVD process on both graphite and CMC materials. Coatings were uniform, exhibited excellent coverage of the samples and appear high density. ZrB_2-SiC multilayer coatings were obtained with good interfacial characteristics.

The next phase is to conduct mechanical and environmental testing of both single and multilayer coatings to determine performance characteristics. Coating processes can then be scaled up from the existing small-scale plant to demonstrative component geometries and application specific substrate materials. In the opposing aspect, additional works are required on the nano-scale to develop a process capable of providing a uniform coating on individual fibres and fibre fabric preforms.

Figure 1: SEM micrographs of ZrB2 coating surface and multilayer ZrB2-SiC cross-section, (note lower micrograph 'layers' from top-bottom are CMC composite, SiC and ZrB_2)