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LIQUID FEEDSTOCK PLASMA SPRAYING - AN EMERGING PROCESS FOR ADVANCED THERMAL BARRIER COATINGS

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ABSTRACT

Liquid feedstock plasma spraying (LFPS) involves deposition of ultrafine droplets of suspensions or solution precursors (typically ranging from nano- to sub-micron size) and permits production of coatings with unique microstructures that are promising for advanced thermal barrier coating (TBC) applications. This paper reviews the recent progress and accomplishments arising from efforts devoted to development of high performance TBCs using the LFPS approach. Advancements in both suspension plasma spraying (SPS) and solution precursor plasma spraying (SPPS), which constitute the two main variants of LFPS, are presented. Results illustrating the different types of the microstructures that can be realized, depicting the correlation between coating microstructure and thermal conductivity, as well as demonstrating the enhancement in functional performances/lifetime possible compared to conventional powder-based coatings, are briefly summarized. TBCs with varied architectures and chemistries, besides the conventional single 8wt. % yttria stabilized zirconia insulating ceramic layer, are specifically highlighted.

INTRODUCTION

Increased functional and environmental demands on today's gas turbines require improved TBCs that are capable of withstanding the higher operating temperatures necessary to meet the incessant drive towards enhanced process efficiency. A 1% increase in engine efficiency of a medium sized power plant of 300 MW results in estimated savings of more than $2 M/year in fuel costs and approximately 25 000 t/year reductions in CO_2 emissions (Oechsner, 2012). Consequently, even small improvements on the above front result in huge benefits to both end-users and environment. However, such advanced TBCs demand new morphologies/microstructures and/or new materials and has constituted the focus of several recent research efforts.

Conventional TBCs have been typically composed of a duplex material system, comprising a ceramic top coat and an intermetallic bond coat, deposited over a suitable high-temperature capable Ni-based superalloy. The function of the topcoat is to provide thermal insulation while the role of the bond coat is to impart oxidation/corrosion protection besides enhanced adhesion of the topcoat to the metallic substrate. Yttria stabilised zirconia (YSZ) is the most commonly used topcoat material due to its low thermal conductivity, high sintering and erosion resistance, relatively high coefficient of thermal expansion and good fracture toughness, to go with its high temperature stability.

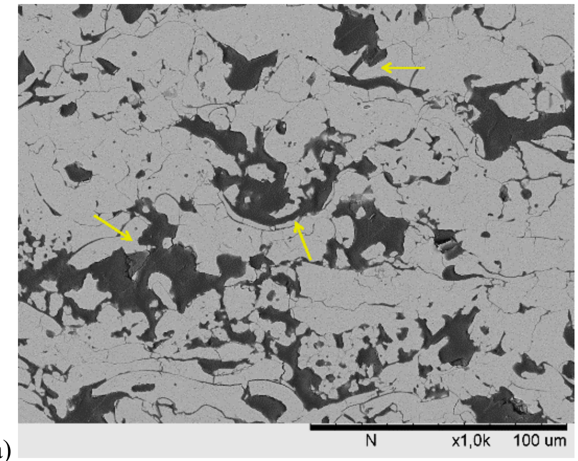
The life-time of a TBC is primary related to its ability to survive the harsh working conditions prevailing in an engine that requires it to resist severe thermal cycling for long periods of time. Although thermal barrier systems exhibit varied failure mechanisms depending upon the operating conditions, a strain tolerant ceramic layer is known to provide excellent thermal cycling durability. The TBCs also need to resist sintering in order to preserve the strain tolerance and thermal insulation properties over time (Zhu and Miller, 2000). Both the above characteristics can be controlled through proper design of the coating microstructure. It has been found that coatings with large globular pores and connected cracks through the coating microstructure can potentially yield both strain tolerance and low thermal conductivity (Gupta, 2015). One of the methods to produce such coatings is by atmospheric plasma spraying (APS) employing a mixture of a ceramic material and a pore former as feedstock (Curry et al., 2013). Although early TBC research had suggested that an optimum level of porosity in the ceramic layer can ensure good strain tolerance of the coating without promoting excessive bond coat degradation, the microstructures that are now acknowledged to yield best strain tolerance are those with a columnar structure or high density of vertical cracks which ensure better compliance of the coating with the metallic substrate under cyclic thermal loads (Liu et al., 2007). The Electron Beam Physical Vapour Deposition (EB-PVD) process is commonly used today to produce columnar TBCs but the high cost of the process and high effective thermal conductivity of the deposited coatings are significant drawbacks (Feuerstein et al., 2008). The more recently developed spraying processes that use a liquid feedstock instead of a conventional powder have demonstrated great potential to produce TBCs with a columnar or vertically cracked microstructure (Kassner et al., 2008). As these coatings are also characterized by high porosity, their thermal conductivity is typically lower than the thermal conductivity of the state-of-art APS and EB-PVD coatings, thereby providing an added benefit (Curry et al., 2014).

The higher operating temperatures essential to enhance engine efficiency also demand new materials for TBCs. Materials such as pyrochlores, perovskites, rare earth garnets etc., have been explored in recent times and found to be capable of withstanding temperatures above 1200 °C (which is the upper limit of the current YSZ TBCs) (Vassen et al., 2009). Although these materials usually exhibit lower toughness and thermal expansion coefficient as compared to YSZ, this provides encouragement to explore multi-layer TBC architectures, with the different layers synergistically combining to fulfil all the requirements of a durable, low thermal conductivity protection system for high temperature operation.

This article briefly highlights the activities in the field of advanced TBCs undertaken by the authors, with use of porosity formers and liquid feedstock to engineer desired microstructures. Both main variants of the LFPS approach, namely suspension plasma spraying (SPS) and solution precursor plasma spraying (SPPS) are discussed. Novel multi-layer architectures involving new TBC materials are also presented.

HIGH PERFORMANCE STRAIN TOLERANT TBC'S BY APS

Thermal sprayed coating microstructures are inherently highly heterogeneous, consisting of distinct features such as pores and cracks of different sizes. The size and shape of these features determines the coating's thermal and mechanical properties and also significantly influences the service lifetime of these coatings (Gupta, 2015). Therefore, in order to achieve a high performance TBC with low thermal conductivity, high strain tolerance and long lifetime, optimization of the coating microstructure is essential. Determination of parametric impact on coating microstructure, as well as optimization of the spray conditions, is usually accomplished through a design of experiments approach. However, this still demands significant experimentation, both in terms of spraying and characterization, and does not necessarily provide fundamental understanding. On the other hand, simulation techniques are advantageous to quantify microstructure-property relationships as well as to develop and analyse new coating designs.

(a)


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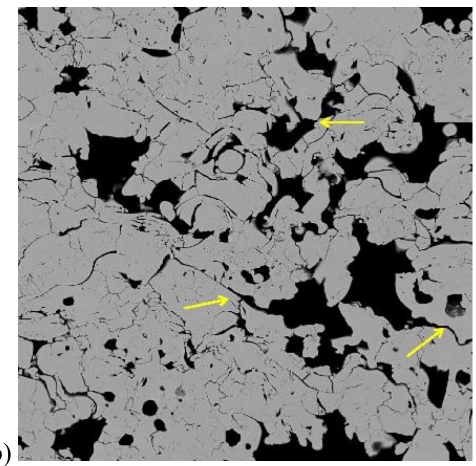


Figure 1. High performance topcoat microstructure achieved by (a) using pore formers in feedstock, and (b) optimising spray parameters (the image represents an area of 180 µm x 180 µm) (Gupta et al., 2013b)

Previous work from this group has shown that coatings with large globular pores and connected cracks through the coating microstructure result in improved strain tolerance and low thermal conductivity (Gupta et al., 2013a). One method to produce such coatings is by conventional APS, using a feedstock powder that is a mixture of a ceramic material and a pore former. This is a promising way of ensuring that the thermal conductivity is low (due to the ceramic material and because of the presence of big pores generated by the pore former) and the coating cohesion is good, since standard spray parameters can be used for spraying the TBC (Curry et al., 2013). The high porosity (around 25%) provides enhanced strain tolerance of the coating. Yet another method to produce similar microstructures is to dispense with the pore former but employ spray parameters with very low power and long spray distance to promote generation of highly porous coatings (Gupta et al., 2013b). However, the deposition efficiency is considerably reduced in this case due to only partial melting of the particles during spraying. Figure 1 shows the microstructures using these two methods where the large globular pores with connected cracks are indicated by arrows.

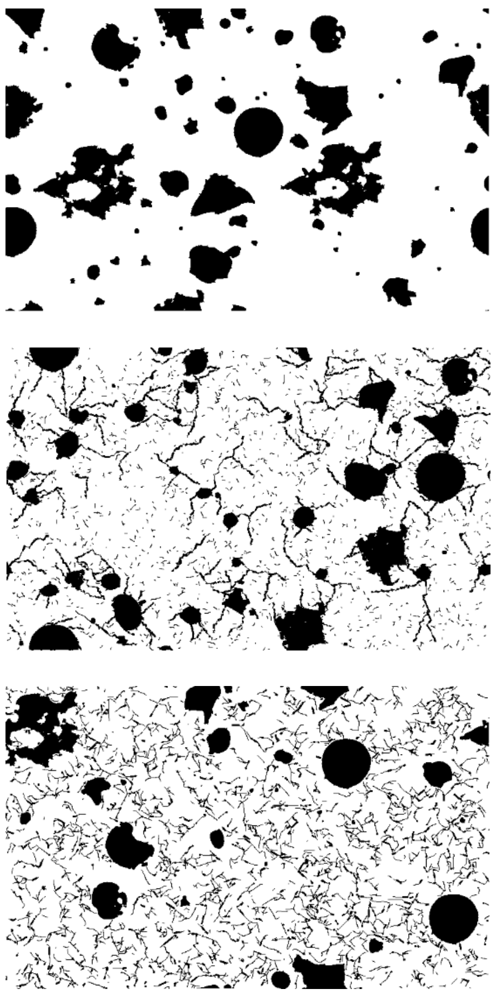


Figure 2. Microstructure images created artificially by modelling representing (a) only pores, (b) free cracks with pores, and (c) connected cracks with pores (Gupta et al., 2013a)

A fundamental understanding of the influence of large globular pores and connected cracks on thermal-mechanical properties of the coating can be achieved with the help of modelling. This can be done by artificially separating the different microstructural features and analysing their individual influence on coating properties. Figure 2 shows the microstructure images created artificially with a microstructure generator modelling tool representing only pores, free cracks with pores, and connected cracks with pores. These images represent a total coating porosity similar to that depicted in Fig. 1. These images were analysed by finite element modelling and it was found that the image shown in Fig. 2(c) results in lowest thermal conductivity and highest Young's modulus as compared to the other two images (Gupta et al., 2013a). These results show that large globular pores with connected cracks are essential for a high performance TBC, and merely increasing porosity by introducing large pores may not be adequate to ensure superior coating performance.

SUSPENSION PLASMA SPRAYING

The limitation of minimum particle size in conventional APS process employing powder feedstock has motivated the development of new plasma spray approaches based on using liquid feedstock in the form of either suspensions or solution precursors. The suspensions typically used in SPS are either based on water or an organic solvent, with the powder particles being in the nano- or sub-micrometric size range.

It is well known that the TBC microstructure has a major bearing on its functional properties. As the particles comprising the suspensions are much smaller than in case of conventional powder feedstock, the mechanisms that control microstructure formation in SPS are more complex than in APS (Fauchais et al., 2006). Fauchais et al. (2008) observed that coating formation in SPS is related to the generation of very fine droplets due to atomization or fragmentation of the suspension after injection, resulting in small in flight particles once the solvent has evaporated. The particles follow the gas stream's trajectory, stick on the side of the asperities on the substrate surface to enable both lateral and vertical growth, and as spraying proceeds, they contribute solely to vertical growth of the coating. This complex interplay can results in the columnar-like microstructure under specific conditions of spraying (VanEvery et al., 2011). The deflection of the particle on impact depends on the Stokes number. Berghaus et al. (2005) have noted that the momentum of the particle at impact is crucially important and, thus, the particle velocity, particle size, and material density are among the factors that strongly influence microstructure formation, including pore shape and size. Ganvir et al. (2015) revealed the strong dependence of particles' in flight characteristics on the spray parameters which, in turn, influence coating formation and microstructure. Four distinct types of microstructures were identified and further correlated with the spray parameters used for deposition. Apart from the vertically cracked TBCs, the columnar microstructures were classified as porous, feathery and columnar (Fig. 3) (Ganvir et al., 2015). In Table 1, the key spray parameters that influence coating formation and microstructure are presented (Ganvir et al., 2015).

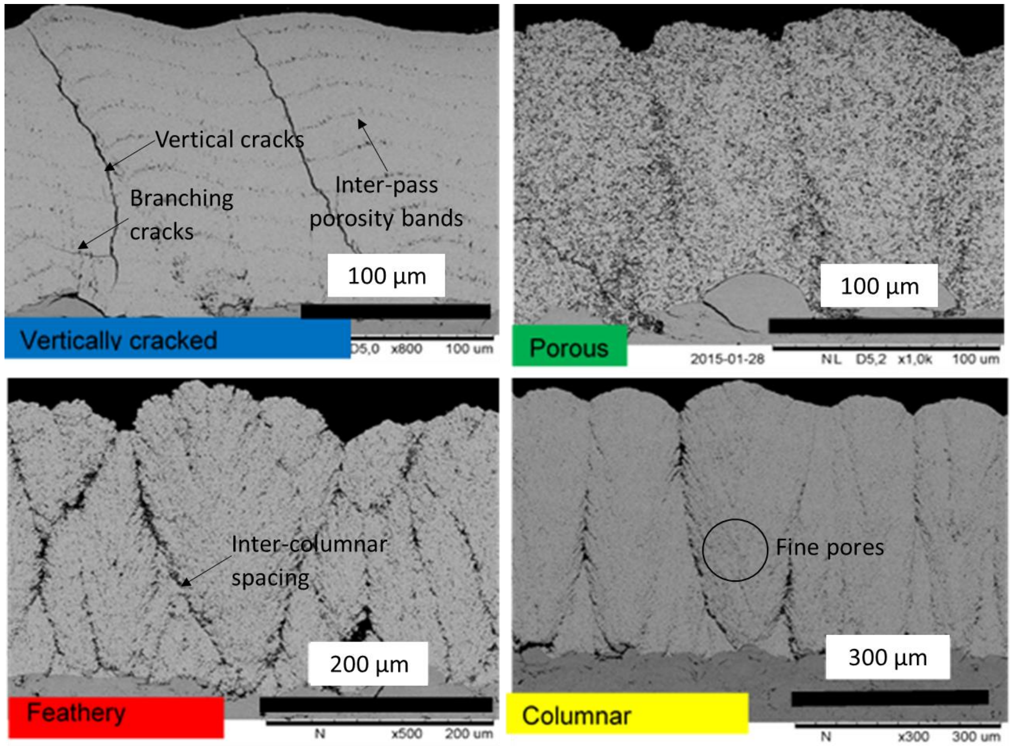


Figure 3. Typical microstructures produced by SPS (Ganvir et al., 2015)

On a macro-scale, the SPS coatings exhibit different microstructural features (coarse porosity) such as: vertical cracks, spacing between columns (inter-columnar spacing), inter-pass porosity bands, branching cracks etc.; whereas at a micro-scale, coatings show features such as fine pores (interconnected or independent). These different features are marked in Fig. 3.

Table 1. Influence of process parameters on microstructure formation (Ganvir et al., 2015)

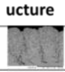
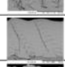
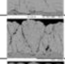
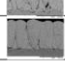
Coating Name	Microstr-ucture	Gas Flow	Spray distance	Surface speed	Feed rate	Current
Porous		Low	Low	High	High	Low
Vertically cracked		High	Low	Low	High	High
Feathery		High	High	Low	Low	High
Columnar		Medium	Medium	Medium	Medium	Medium

Figure 4 shows a comparative distribution of fine pores (<1μm^2) and coarse pores (>1μm^2) in five samples sprayed on similar substrate specimens and with identical feedstock materials but with different spray parameters. The microstructural features such as the extent of fine and coarse porosity, column density etc., are governed by the deposition conditions employed (Ganvir et al., 2016). These features also undergo changes upon prolonged thermal exposure.

(a)

(b)

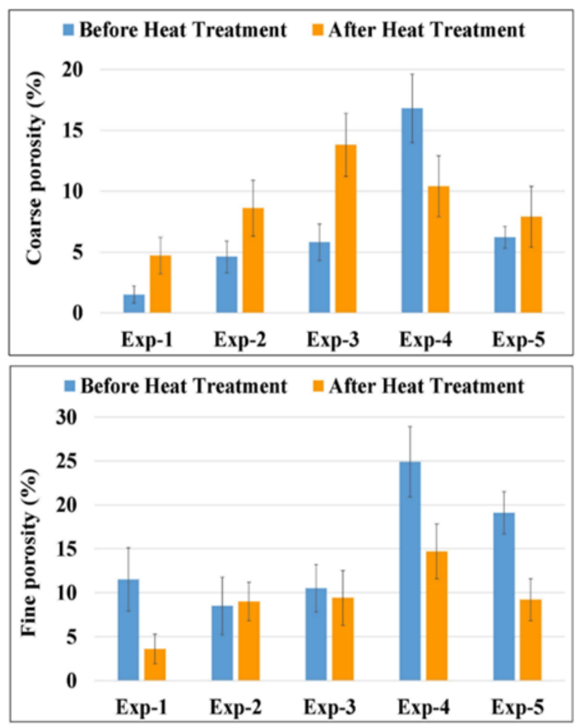


Figure 4. Quantification of a) Coarse porosity and b) fine porosity by image analysis technique before and after isothermal heat treatment (Argon at 1150°C for 200 h) (Ganvir et al., 2016)

The microstructural features in a TBC also influence its thermal properties. In Fig. 5, the thermal conductivity of the various samples characterized in Fig. 4 is presented. The thermal conductivity was measured by Laser Flash Analysis and found to be lowest in coatings exhibiting highest porosity. In certain samples these values were found to change significantly after heat treatment (Ganvir et al., 2016). The process parameters employed for producing the samples presented in Fig. 4 and 5 are presented in Table 2 (Ganvir et al., 2016).

Apart from the spray parameters influencing the coating microstructure as discussed above, Curry et al. (2015) have shown that bond coat roughness, too, has a direct influence on the column density. In Fig. 6, it can be seen that, as the roughness of the bond coat increases, the columns' density in TBCs decreases. All four samples presented in Fig. 6 were sprayed in similar conditions, with only the bond coat roughness being altered in different ways as indicated in the figure.

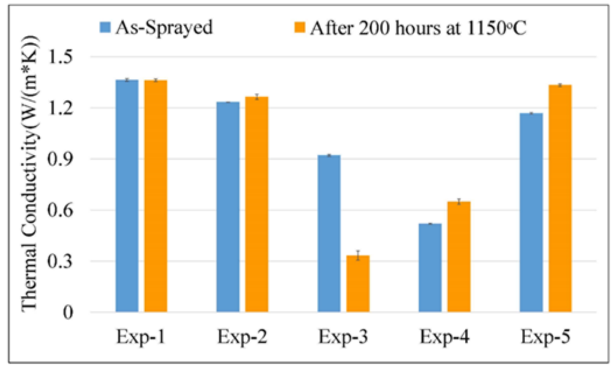


Figure 5. Thermal conductivity results of various SPS coatings before and after heat treatment (Ganvir et al., 2016)

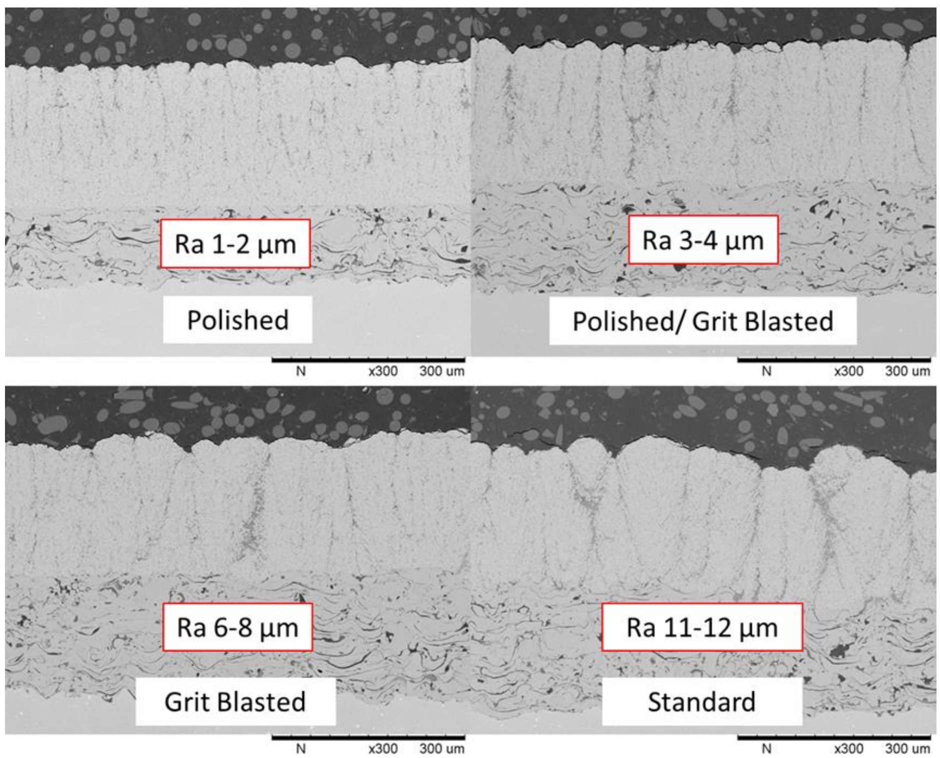


Figure 6. Influence of bond coat roughness on column density (Substrate: Hastalloy X, Bond coat: AMDRY 386, sprayed by APS, F4 gun, topcoat: 8YSZ suspension, 10wt.% solid load, sprayed with Mettech Axial III gun) (Curry et al., 2015)

The chemistry of the bond coat as well as the particle size of the bond coat powder feedstock also play an important role in determining the functional properties of SPS TBCs. As illustrated in Fig. 7, when different compositions and particle sizes were used for spraying the bond coats (with all other spray parameters, ceramic top coat material and test conditions remaining identical), the thermal cyclic life of the samples was found to vary significantly (Markocsan et al., 2015).

Table 2: Process parameters used for production of all five types of coatings presented in Fig. 4 & 5 (Ganvir et al., 2016)

Process parameter	Specimen Nomenclature				
	Exp1	Exp2	Exp3	Exp4	Exp5
Spray distance (mm)	75	50	100	100	100
Surface speed (cm/s)	145.5	75	75	216	216
Suspension feed rate (mL/min)	70	45	45	100	45
Total gas flow rate (L/min)	250	200	300	300	200
Total power during spray (kW)	125	101	124	124	116
Total Enthalpy during spray (kJ)	13	11.2	12.5	12.5	11.2

NEW MATERIALS AND MULTILAYERED SYSTEMS

Due to the known drawbacks of YSZ above 1200 °C, such as decomposition into high yttria and low yttria phases, significant sintering etc., the search for new TBC materials without compromising requirements such as sintering resistance, phase stability, thermal conductivity, oxidation resistance and CMAS penetration resistance has been a subject of considerable research interest. Pyrochlores are promising for fulfilling the above requirements at higher temperatures (Stöver et al., 2004, Schmitt et al., 2014, Vassen et al., 2000, Wu et al., 2002). Among the pyrochlores, gadolinium zirconate ($Gd_2Zr_2O_7$) and lanthanum zirconate ($La_2Zr_2O_7$) are interesting candidates, although the latter is difficult to process due to the tendency for La_2O_3 to evaporate and result in loss of desired stoichiometry (Xu et al., 2010, Mauer et al., 2013). Gadolinium zirconate (GZ) has excellent phase stability and lower bulk thermal conductivity than YSZ. However, it has a lower fracture toughness (Choi et al., 2005, Bakan et al., 2014, Zhong et al., 2014) and also a tendency to react with and degrade the alumina that forms on the bond coat as the protective thermally grown oxide (TGO) above 1200 °C (Leckie et al., 2005). In order to overcome these drawbacks, a multilayered approach with GZ on top of YSZ has been proposed (Zhong et al., 2015, Lee et al., 2014, Bakan et al., 2015). The functional performance of such a system has been evaluated and compared with single layer 8YSZ coatings (Mahade et al., 2015, Mahade et al., 2016a, Mahade et al., 2016b). Both coating systems were deposited using SPS. Additionally, a triple layer TBC comprising a relatively denser 30 µm thick GZ layer on top of a GZ/YSZ TBC was deposited by SPS in order to impart better erosion and CMAS attack resistance. The three different coating architectures investigated are shown in Fig. 8.

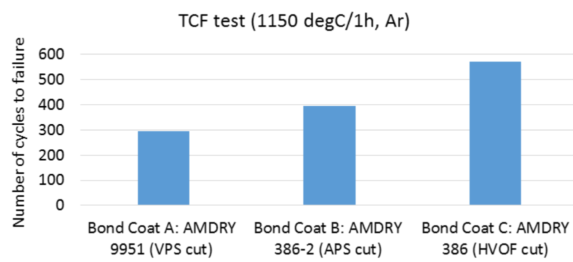


Figure 7. TCF life time of various 8YSZ SPS TBCs sprayed on bond coats with varying chemistry and particle size

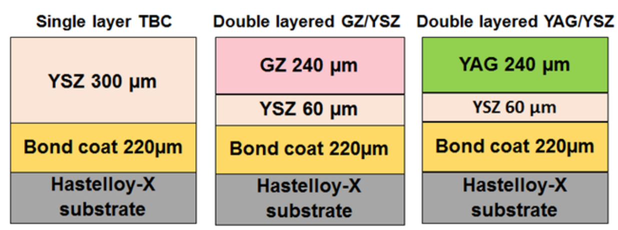


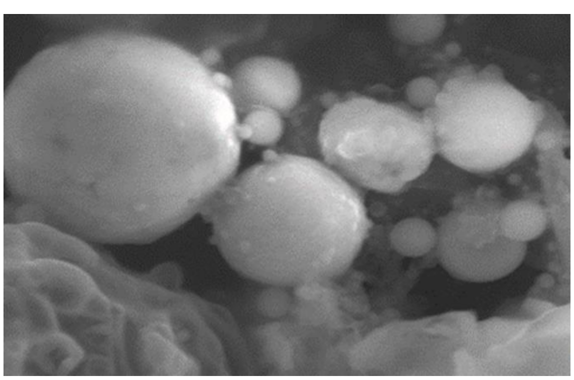
Figure 8: Architectures of the three different TBCs studied

Thermal conductivity, thermal cyclic life and erosion resistance of the three different TBC designs were compared. The as-sprayed TBCs were subjected to thermal cyclic fatigue test at 1100°C and 1200°C. Also, the thermal conductivity of as-sprayed TBCs was measured in the temperature range of 25°C to 1000°C. The results revealed that the GZ based multi-layered TBCs had a higher thermal cyclic life and lower thermal conductivity compared to single layer YSZ TBC. Erosion tests at room temperature were also carried out on the TBCs. Among the as-sprayed TBCs, double layer GZ/YSZ exhibited lowest erosion resistance. The triple layer GZ dense/GZ/YSZ TBC had a slightly better erosion resistance than the double layer TBC due to the presence of relatively denser GZ on top. The study showed that columnar microstructure to mimic the EB-PVD process can be created in both single and double layer TBCs by SPS, and a dense third layer also deposited on top of the columnar coatings.

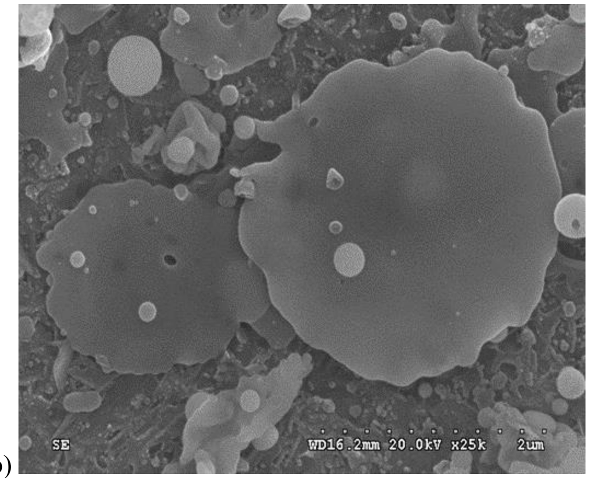
SOLUTION PRECURSOR SPRAYED TBC

Similar to the SPS technique discussed above, the SPPS method has also been the subject of considerable research interest in recent times due to the several inherent advantages that this route offers. The SPPS method relies on the use of suitable solution precursors that generate particles of the desired coating material *in situ* and, thereby, provides the added benefit over SPS of obviating the need for expensive nano- or sub-micron sized powder feedstock. While this is a major attraction, the SPPS process is also more complex to control / optimize and has consequently not been investigated as widely as the SPS. Prior work has shown the SPPS coatings to possess interesting intrinsic features like vertical cracks, homogenous fine pore structure, splats that are an order of magnitude smaller than in conventional APS etc. as well as greater durability under thermal cycling conditions (Gell et al., 2008, Fauchais and Montavan, 2010).

As in case of SPS, the relevant properties of SPPS TBCs in terms of strain tolerance, thermal conductivity, longevity under thermal cycling conditions etc. are governed by its microstructural design. However, the ability to manipulate the TBC microstructure through appropriate control of spraying conditions is intimately dependent on a complete understanding of the mechanism responsible for coating formation. The short residence times (typically of the order of few milliseconds) that are available for the rapid transformation of the precursor solution into a coating, and the inappropriateness of the tools usually used to investigate in flight particles in conventional plasma spraying to diagnose SPPS, had hampered such an understanding for long. However, studies have now revealed that the properties of SPPS coatings can be correlated with the *in situ* particle generation and the subsequent formation of splats when these particles impact the substrate (Sivakumar et al., 2011). It has further been realized that, apart from the particles formed in flight, any unpyrolyzed precursor impacting the preheated substrate is another crucial factor influencing the microstructure of the coating formed.



(a)



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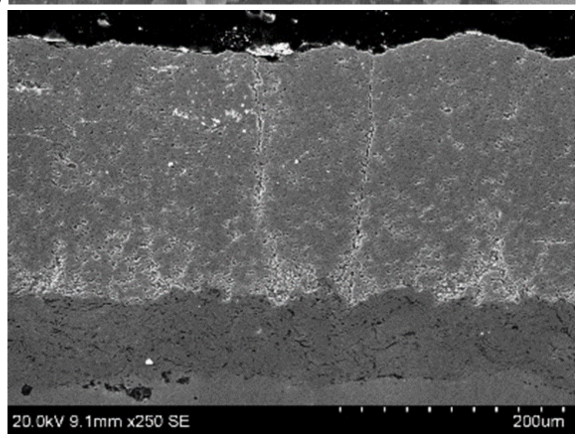

(c)

Figure 9. SPPS characteristics: (a) typical in flight generated particles (b) typical single particle splat and (c) vertically cracked microstructure obtained under appropriate processing conditions during SPPS (Sivakumar et al., 2011, Sivakumar et al., 2014)

In SPPS coatings, the presence of through-thickness vertical cracks in the deposited TBCs is critical for enhancing their strain tolerance and, thereby, their performance and durability. A microstructure with high segmented crack density (number of vertically aligned cracks per mm across a defined cross section) and moderate porosity has been reported to yield good thermal cycling performance. It has also been proposed that evolution of the vertical cracks can be attributed to pyrolytic stresses resulting from precursor decomposition at the substrate. Based on the above understanding of the SPPS process, it has been demonstrated that varying the solution precursor flow rate provides an ideal pathway for controlling the coating microstructure in SPPS YSZ coatings. Since the pyrolytic stress can vary depending upon the amount of unpyrolyzed precursor incorporated in the coating, and the plasma heat input available for complete/partial decomposition of the precursor, suitable control of the spray conditions process variables can be exploited to ensure vertical crack formation in SPPS YSZ coatings. Figure 9 illustrates typical micrographs of particles generated in flight and splats formed upon impact of these particles with the substrate. A vertically cracked YSZ coating obtained by controlling the spray conditions is also shown. The improved understanding of the process has laid down the foundation to explain the associated mechanisms (Sivakumar et al., 2014) as well as further expand the utility of the SPPS process (Sivakumar et al., 2013).

The enhanced appreciation of the process emerging from the above studies provides an ideal foundation to exploit the wide-ranging benefits of the SPPS technique and its versatility. Exciting prospects for hybrid processing, combining the SPPS method with conventional powder-based plasma spraying, to yield novel microstructures and superior properties have already been shown (Joshi et al., 2014).

CONCLUSIONS

Solution-based plasma spraying is poised to open new frontiers in surface engineering. The solution routes, comprising solution precursor plasma spraying (SPPS) and suspension plasma spraying (SPS), have been the subject of considerable research interest in recent times in recognition of the fact that they offer significant advantages such as generation of nanostructured coatings, permitting better control over coating chemistry and yielding interesting microstructural features like columnar structures / vertical cracks, nano-sized pores, fine splats etc. The utility of the above techniques to conveniently enable deposition of coatings with various architectures and utilizing new chemistries has already been demonstrated and holds particular promise for deposition of advanced TBC systems.

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