



Vapor Phase Deposition Using Plasma Spray-PVD™

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Plasma spray—physical vapor deposition (PS-PVD) is a low pressure plasma spray technology to deposit coatings out of the vapor phase. PS-PVD is a part of the family of new hybrid processes recently developed by Sulzer Metco AG (Switzerland) on the basis of the well-established low pressure plasma spraying (LPPS) technology. Included in this new process family are plasma spray—chemical vapor deposition (PS-CVD) and plasma spray—thin film (PS-TF) processes. In comparison to conventional vacuum plasma spraying and LPPS, these new processes use a high energy plasma gun operated at a work pressure below 2 mbar. This leads to unconventional plasma jet characteristics which can be used to obtain specific and unique coatings. An important new feature of PS-PVD is the possibility to deposit a coating not only by melting the feed stock material which builds up a layer from liquid splats, but also by vaporizing the injected material. Therefore, the PS-PVD process fills the gap between the conventional PVD technologies and standard thermal spray processes. The possibility to vaporize feedstock material and to produce layers out of the vapor phase results in new and unique coating microstructures. The properties of such coatings are superior to those of thermal spray and EB-PVD coatings. This paper reports on the progress made at Sulzer Metco to develop functional coatings build up from vapor phase of oxide ceramics and metals.

Keywords ceramic oxide layers, coatings for gas turbine components, diagnostics and control, TBC top-coats, thermal cycling, thermal properties, TS coating process

1. Introduction

All thermal spray processes have in common that the coating buildup is done by splats of molten or semi molten coating material. In contrast to the thermal spray technology, the processes of physical vapor deposition (PVD) build up a coating out of the vapor phase at low ambient pressure ($\sim 10^{-4}$ mbar) (Ref 1). The hot coating material is not deposited onto a cold surface where droplets solidify rapidly but condenses on the substrate out of the vapor. This leads to coatings with characteristics not possible to achieve with any thermal spray process so far. PVD coatings can be very homogeneous, thin, dense, hard, and gas tight or can have a special designed microstructure.

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The columnar microstructure of yttria-stabilized zirconia (YSZ) deposited with electron beam physical vapor deposition (EB-PVD), for example, is especially suited for high strain tolerant thermal barrier coatings (TBCs). The drawbacks of conventional PVD processes compared to thermal spray are high investment costs and low deposition rates. This is why they are mainly applied for thin coatings in mass production or on top of valuable components like turbine vanes and blades for aircraft engines. Furthermore, conventional PVD processes can only cover surfaces which are in direct line of sight to the coating source. Therefore, components with undercuts or complex geometries are very difficult to coat homogeneously. If it would be possible to coat out of the vapor phase using a thermal spray process with high coating quality, high deposition rates and comparable low investment costs, thermal spraying could expand its field of application with advantages in coating performance and price. This would open a whole new market and future growth perspective. In that perspective, Sulzer Metco has been successful by modifying a standard low pressure plasma spraying (LPPS) process into a vapor-capable deposition process which is called PS-PVD. This process is also capable to coat areas which are not in the line of sight of the coating source. This makes the coverage of parts with complex geometries such as multiple turbine air foils possible.

2. PS-PVD Technology

2.1 System Equipment

The PS-PVD process is based on the ChamPro™ technology of Sulzer Metco which comprises all those thermal

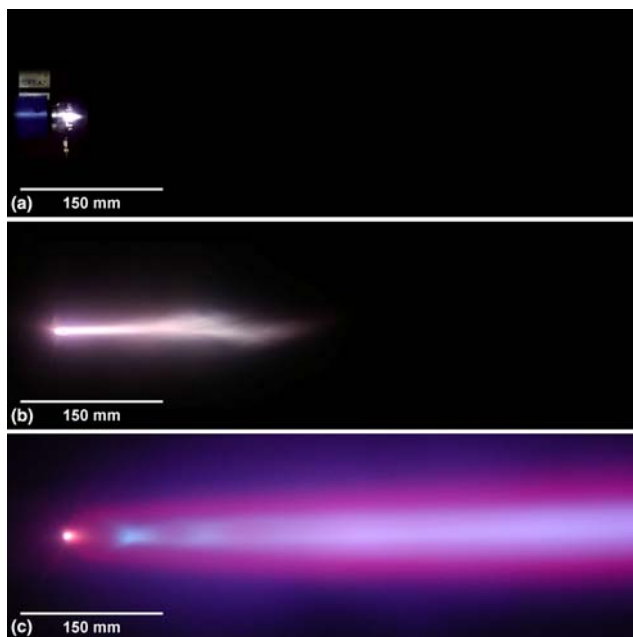


Fig. 1 Images of the plasma jets expanding at different pressures (a) 950 mbar (APS), (b) 50 mbar (VPS/LPPS), and (c) 1 mbar (PS-PVD)

spray processes performing under a defined and controlled atmosphere like LPPS, vacuum plasma spraying (VPS), and low vacuum plasma spray (LVPS) (Ref 2). These processes operate under reduced pressure conditions, typically in an argon gas atmosphere. The inert atmosphere is used to minimize the deteriorating influence of oxygen intrusion into the spray plume and helps to avoid oxidation or contamination of the sprayed deposits. The common working pressure ranges of the standard vacuum processes are between 50 and 200 mbar and allow the deposition of layers having a typical thickness range between 20 μm and 2 mm. The reduced pressure compared to atmospheric conditions enlarges the plasma plume from 50 up to 500 mm in length and from 10 up to 40 mm in diameter. The larger plume results in a bigger spray spot. Therefore, particle velocities and temperatures are more homogeneously distributed over the cross section of the plume (Ref 3). This helps coating complex geometries with a homogeneous coating thickness distribution, in particular turbine parts.

The PS-PVD process operates at even more reduced pressures down to 0.5–2 mbar. Under these conditions, the properties of the plasma jet changes substantially (Ref 4). The plasma plume expands to more than 2 m long and 200–400 mm in diameter (Ref 5, 6). In Fig. 1, the effect of the pressure on the plasma length for air plasma spray (APS), VPS/LPPS, and PS-PVD is shown.

A modified single cathode vacuum plasma gun, the Sulzer Metco O3CP gun, is used which allows a high total gas flow of up to 200 SLPM and high power levels up to 180 kW (3000 A). The gun can be run with a two- or fourfold internal powder injection. Pre-heating and cleaning of the substrate can be applied using the plasma

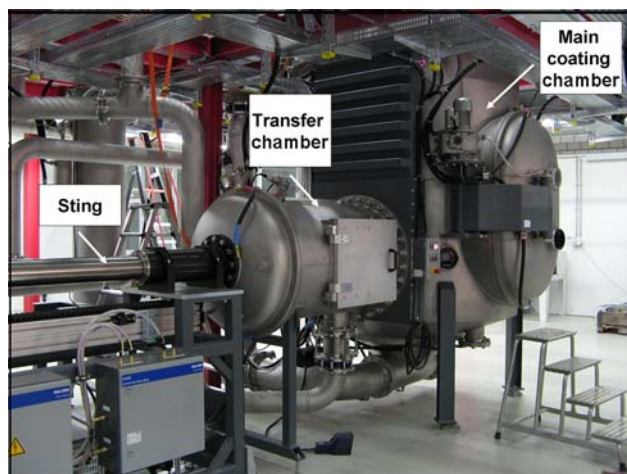


Fig. 2 PS-PVD system with vertical process chamber and horizontal sting with transfer chamber

or an integrated transferred arc processing to optimize the coating adhesion. A horizontal water-cooled sting manipulator with a transfer chamber allows short cycle time and the handling of various substrate dimensions and geometries. The setup of the new PS-PVD prototype system at Sulzer Metco is shown in Fig. 2.

Even though the PS-PVD work pressure (~ 1 mbar) is still much higher than the work pressure used for conventional PVD processes ($\sim 10^{-4}$ mbar), the combination of a low pressure and a high energy level of the plasma gun enables a defined evaporation of the injected powder material and allows a controlled deposition out of the vapor phase. In EB-PVD, the transport of the vaporized coating material toward the substrate is a diffusion process with limited throughput and coating growth rates. In contrast to that, PS-PVD incorporates the vaporized coating material in a hot supersonic gas stream (~ 2000 m/s at 1 mbar and 6000–10,000 K). This leads to high growth rates and the possibility to coat undercuts and areas which are not in the line of sight.

2.2 Process Conditions and Characterization

In order to monitor and characterize the evaporation and deposition process, several diagnostics are installed on the PS-PVD system as shown in Fig. 3.

Standard diagnostic tools such as the DPV-2000/Acuraspray (Tecnar Automation Ltd., Canada) are usually used to detect the molten particles in-flight. As evaporated particles remain undetected by conventional techniques such as DPV-2000 or CCD (charge-coupled device) cameras, optical emission spectroscopy (OES) is used to determine plasma properties and also to characterize the emission of evaporated particles inside the plasma jet. A [Pyroview 380G] infrared camera (DIAS Infrared Systems, Dresden, Germany), an [IGA 5] infrared—pyrometer (Impac, Frankfurt, Germany), and [Type K and N] thermocouples (Thermocoax, Flers Cedex, France) are used to measure and control the substrate temperature during the

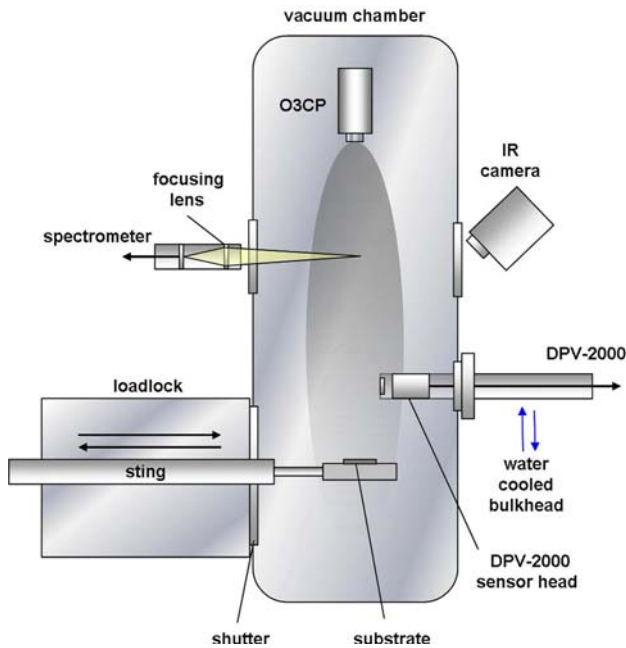


Fig. 3 Schematic figure of the diagnostics used

coating process. Finally, a fast [Pixelfly] CCD Camera (PCO, Kelheim, Germany) with a tele-zoom lens is used to monitor the injection of the powder at the nozzle exit and also to analyze the stream of the supersonic plasma jet around the coated substrate during the coating process. Experimental investigations of the plasma jet as well as the study of spray particle properties have demonstrated the special characteristics of this process which are described in detail elsewhere (Ref 7-9).

3. Applications for Vapor Phase Deposition

The main focus of this work using PS-PVD is the vapor deposition of YSZ to develop a high temperature-resistant TBC similar to EB-PVD coatings for turbine applications. The main interest is to deposit a coating having a columnar microstructure capable of high strain tolerance. Since vapor deposition of metallic coatings is also of high interest, initial investigations have been performed using this new technology. Such metallic coatings could be used for high-quality bond coats in turbine components or other applications. The main challenge is to have a suitable feedstock material and to find optimal parameters to completely vaporize the coating material. Another important challenge beside the evaporation of coating material is to grow a coating out of the vapor onto the substrate which fulfill the requirements of a high quality bond coat, such as designed chemical composition and suitable microstructure. All developed coatings have been benchmarked with respect to other existing coating solution in the case of turbine parts.

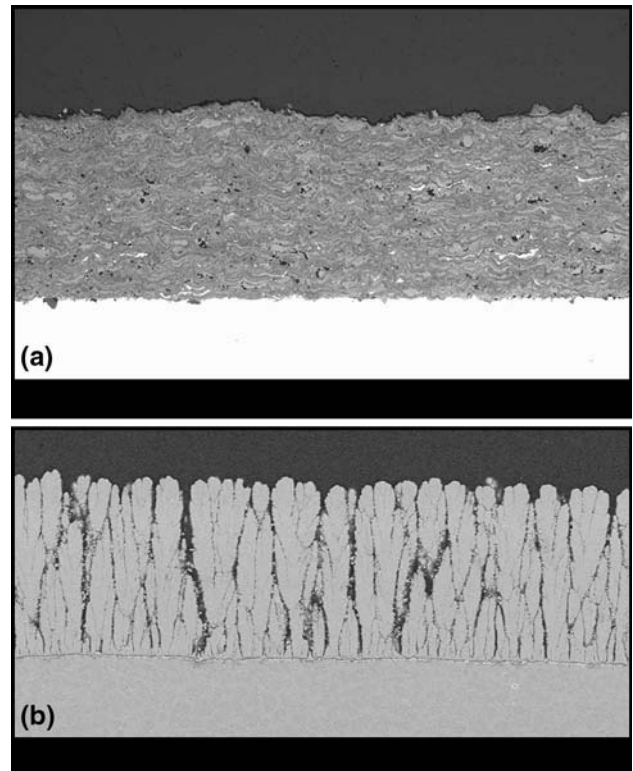


Fig. 4 TBC coating of 7YSZ using different PS-PVD plasma parameters showing (a) splat-like structure, and (b) columnar structure

3.1 TBC with Columnar Microstructure

To ensure the evaporation of the injected coating material, a fine grain sized powder feedstock is used. The 8 wt.% YSZ powder Metco 6700 ($<25\ \mu\text{m}$) has been developed especially for this purpose. With this particular powder and new PS-PVD technique, it is possible to produce EB-PVD like coatings having a columnar microstructure. Analysis of the coating microstructure shows that the application of PS-PVD, even being a thermal spray process, allows the evaporation of the sprayed material to form special coating microstructures similar to EB-PVD coatings. Variations of the microstructures can be produced by varying the process conditions. Figure 4 shows two coatings made with different spray parameters. The type of layer structure can be controlled by the spray parameters, turning it from a splat type layer (see Fig. 4a) over to a porous or finally dense columnar structure. The favorable condition to obtain the columnar structure as shown in Fig. 4(b) occurs using a low powder feed rate, a specific mixture of argon and other plasma gases and having a large spray distance.

Optical emission spectroscopy (OES) was used to verify if specific plasma parameters have a measurable influence on the injected material inside the plasma jet. Figure 5(a) shows the spectrum of the plasma jet with

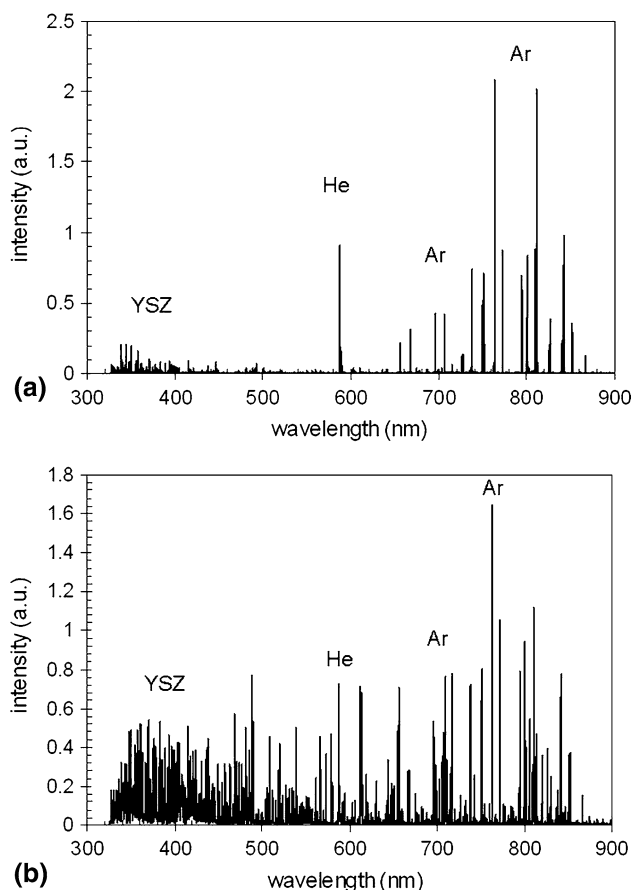


Fig. 5 Optical spectrum of the plasma jet (Ar/He) with 7YSZ corresponding to coatings having (a) no columnar structure and (b) with columnar structure

injected YSZ powder using plasma parameters to produce splat-like coatings. The intensity of the spectral lines coming from the YSZ species is low which shows that only a minor amount of the coating material is evaporated. In Fig. 5(b), the spectrum presents an increased intensity of the spectral YSZ lines which cover the full wavelength bandwidth of the spectrometer (350-900 nm). The spectral lines correspond to the different species composing the YSZ, namely zirconium and yttrium. Here, the injected material could be vaporized which allows the growth of columnar structured coatings.

Typical substrate temperatures during the spray process can vary between 900 and 1100 °C depending on the spray conditions and substrate material. In general, a higher substrate temperature is advantageous for the formation of a well-defined columnar microstructure. The YSZ columns grow perpendicular to the substrate surface. If the coating is applied to rough surfaces, the growing columns interfere with each other and do not build a homogeneous structure. If the coatings are applied on smooth surfaces, the column axes orient themselves parallel. Therefore, the surface roughness of the surface to be coated should not exceed a Ra value of 2 μm to allow a homogeneous structure. The coating deposition out of the vapor onto the substrate

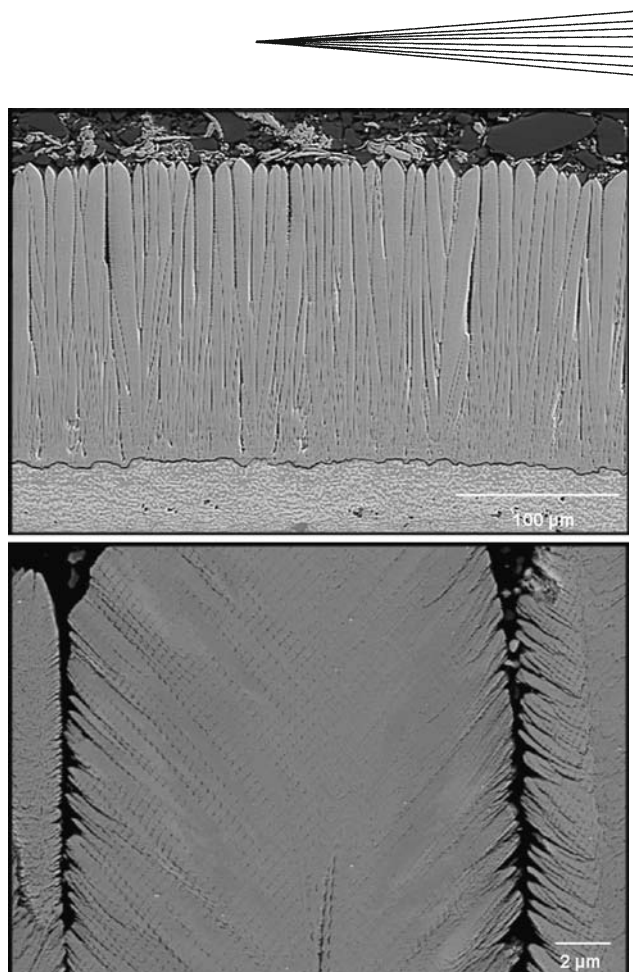


Fig. 6 Typical EB-PVD 7YSZ TBC microstructure in cross section (courtesy of U. Schulz, DLR, Institute of Materials Research, Cologne, Germany)

takes place in the hot center of the plasma plume. Growth rates inside the plasma plume are very fast and can be more than 100 $\mu\text{m}/\text{min}$. To avoid the overheating of the substrate from the high enthalpy of the plasma, the coating build up is obtained by producing a fast relative motion between the plasma jet and the substrate. The fast relative motion also ensures a homogeneous coating temperature distribution of the substrate. Deposition efficiencies of about 30-40% with a typical growth rate of about 0.1-0.2 $\mu\text{m}/\text{pass}$ could be achieved on flat metallic samples. This corresponds to a deposition rate on real parts such as turbine blades in the range of ≥ 10 -20 $\mu\text{m}/\text{min}$ depending on the geometry and size of the substrate.

EB-PVD produced coatings consist of a quite homogeneous columnar structure composed of compact single columns. The bulky structure is only interrupted by a certain amount of inter-columnar gaps and voids between feather arms on the outer shell and a small amount of internal porosity (Ref 10, 11). A typical EB-PVD 7YSZ TBC microstructure in cross section can be seen in Fig. 6. In contrast, PS-PVD produced columns consist of many fine needles with a high defect density and a high amount of internal porosity. A typical PS-PVD columnar structure with a layer thickness of about 250 μm is shown in Fig. 7.

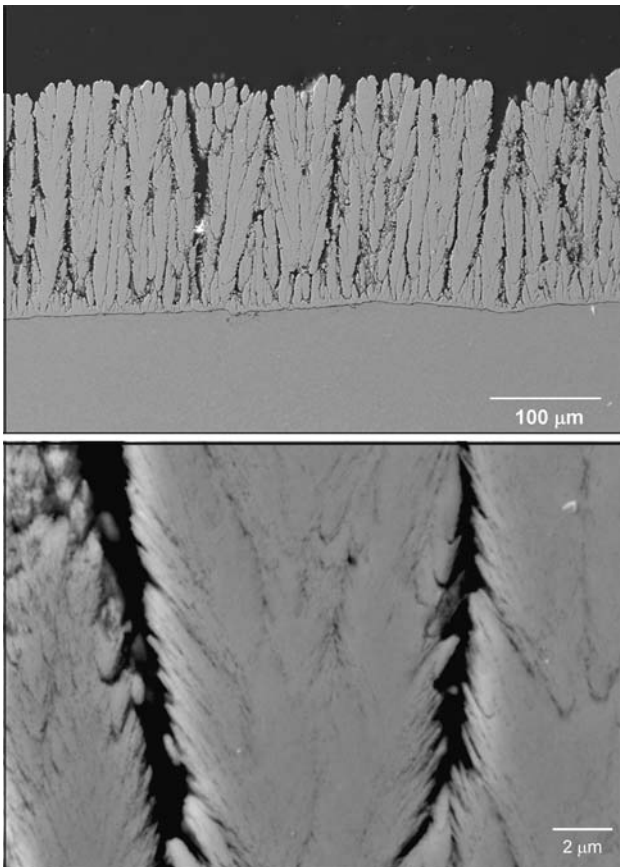


Fig. 7 Columnar TBC top layer deposited with PS-PVD on a MCrAlY bond coat (low picture is a higher magnification of the top picture)

YSZ coatings with a thickness of 150-300 μm have been produced with PS-PVD on metallic test samples (Inconel 718, Rene N5 and Rene 142) having a vacuum plasma-sprayed MCrAlY or an electro-chemical deposited PtAl bond coat to characterize the columnar coatings and to validate the different morphologies. Erosion tests, thermal cycling tests, and measurements of the thermal conductivity have been performed. Erosion resistance was measured using an erosion test rig according to the GE specification (GE: E50TF121) at room temperature. An aluminum oxide powder with a grain size of 40-80 μm is used as erodent and is accelerated at room temperature onto the TBC surface at an angle of $20^\circ (\pm 2^\circ)$ and with a pressure of 2 bar (± 0.25 bar). The tube to accelerate the erodent is 95 mm in length and its standoff distance from the TBC surface was 102 mm. The erosion resistance is measured as the time (in seconds) needed to erode 25 μm of coating. Figure 8 shows the erosion resistance of different PS-PVD microstructures together with APS and EB-PVD coatings as reference. Initial measurements showed low erosion resistance. This can be explained by the fine needle structure of the single columns and the inter-columnar gaps between the columns which give a low mechanical strength to the structure (see Fig. 7). With

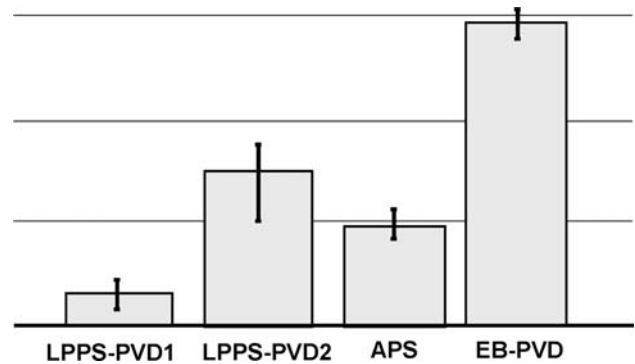


Fig. 8 Erosion-resistance of different PS-PVD structures and APS, EB-PVD reference coatings

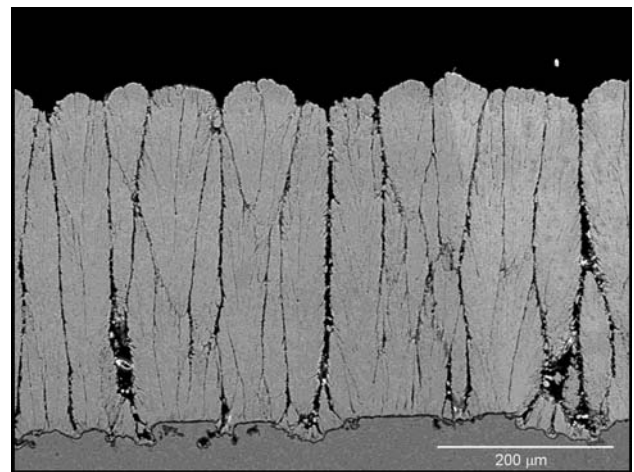


Fig. 9 Optimized columnar TBC structure deposited with PS-PVD on a MCrAlY bond coat

an adequate process and parameter optimization, the density of the structure could be improved and the inter-columnar gaps could be reduced to a minimum. With the resulting improved microstructure, an increased erosion resistance between the value of APS and EB-PVD TBCs have been obtained (PS-PVD2).

A picture of an optimized columnar structure is shown in Fig. 9. Furnace cycling tests (FCT) were undertaken to screen the coating performance in terms of strain tolerance. A standard test cycle on air between 1135 $^\circ\text{C}$ and room temperature was used (Ref 12). The heat up time is 6 min with a hold period of 50 min followed by a cool down to room temperature for 4 min. The hourly cycle is interrupted for a period of 4 h every 24 h to allow water vapor to infiltrate the structure at room temperature and to validate coating spallation. The failure criterion was defined as the time or the number of cycles needed for the onset of coating spallation or buckling of at least 25% of the total coating surface. The PS-PVD columnar structure showed superior results with more than 700 cycles achieved without any failure of the coating. These results have been achieved with substrates having a PtAl

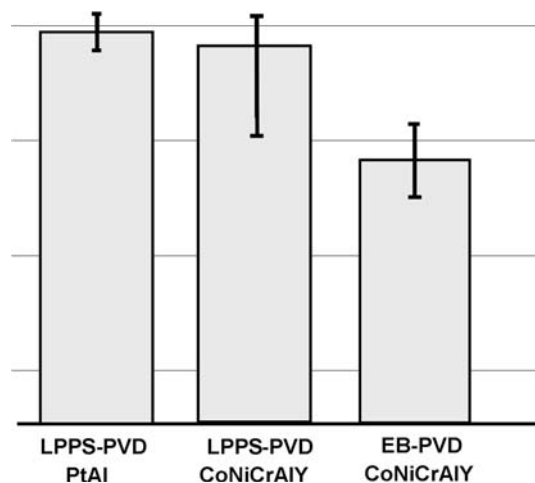


Fig. 10 FCT results of different bond coats with PS-PVD top-coat and EB-PVD reference coatings (all samples had a coating thickness of 150 μm)

bond coat. Substrates with a MCrAlY bond coat also performed at a high level, but showed much more variance and sensitivity to the process parameters, such as pre-heating temperature, surface roughness, and thermally grown oxide formation due to bond coat oxidation. In Fig. 10, FCT results using different bond coats and EB-PVD TBC (with MCrAlY bond coat) reference measurements are shown. It is assumed that a defined pre-oxidation of the bond coat to achieve a thin $\alpha\text{-Al}_2\text{O}_3$ layer before TBC deposition can further increase the good performance of the PS-PVD coated TBCs. Such an approach was successfully verified by other authors (Ref 13-15).

Thermal conductivity measurements of different PS-PVD structures were conducted using the laser flash method (Netzsch Thermal Analysis, Selb, Germany). All measurements showed very good high temperature insulation properties with a thermal conductivity of about 0.8 W/m K at room temperature without any noticeable change up to 1000 $^\circ\text{C}$. The good results obtained can be explained by the high amount of porosity and defects within the PS-PVD microstructure. To demonstrate the ability to cover complex turbine blade geometries with a uniform morphology and coating thickness distribution, real gas turbine blades have been coated using the PS-PVD technology. The coating had clear columnar structures at all substrate locations. Overall, a homogeneous coating thickness of about 150-200 μm was achieved. The thickness was slightly reduced on the suction side with a stronger increase at the leading edge. Even along the fillet region, a columnar structure was deposited, showing a somewhat reduced thickness of about 100-120 μm .

3.2 Metallic Vapor Deposition

The first step toward metallic vapor deposition presented here mainly includes analysis of the standard LPPS process. The difficulty is to determine to what extent a coating is produced from vapor phase or from a dense

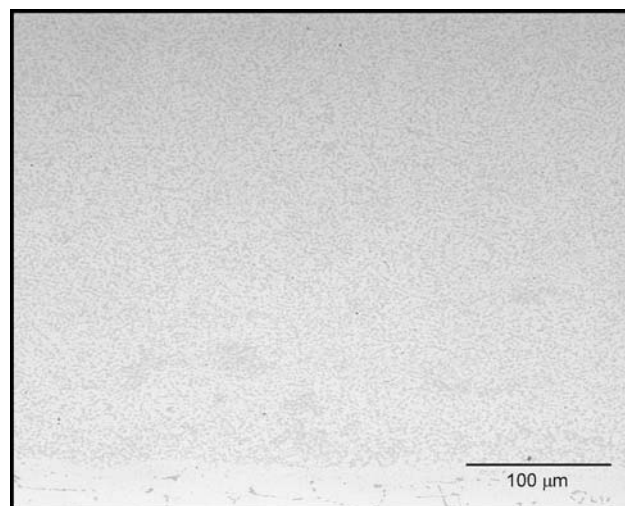


Fig. 11 State of the art MCrAlY bond coat after heat treatment

layer of splats. However, it is assumed that vaporization of a certain amount of coating material is already part of the standard LPPS process. Similar to the YSZ case, OES can be used to detect species in the vapor phase in the plasma jet. These measurements show that denser coatings are obtained when the emission lines of the metallic powder in the vapor phase are higher. Using a fine MCrAlY powder (Sulzer Metco Amdry 365, $<35\ \mu\text{m}$) and standard parameters, the spectrum already contains some emission lines of metal vapor species. The resulting coatings show splat-like microstructures, which vanish during heat treatments, as common for LPPS MCrAlY bond coats. This means that having a minor amount of metal vapor in the plasma plume is not new for VPS. Figure 11 shows the state of the art MCrAlY bond coat after heat treatment.

If the process is controlled to minimize emission lines for metal vapor, the splat-like character of the coating becomes more evident. An optical emission plot and the corresponding microstructure of such a coating are shown in Fig. 12.

If the process is run under PS-PVD conditions, the emission lines corresponding to the metal vapor have clearly higher intensities than the ones running the process under PS conditions. The distinctive metal lines (Ni, Cr) reveal that the powder is much more vaporized. Coatings produced using PS-PVD parameters showing high emission lines of Ni and Cr (see Fig. 13a) result in very dense coatings with no visible oxide layers already in the as-sprayed condition without any heat treatment applied (see Fig. 13b).

Figure 14 shows a detail of the emission lines in the wavelength range between 350 and 550 nm. The measured spectral lines are allocated to single elements, mainly Cr and Ni lines. If the MCrAlY powder would be fully vaporized, additional lines of Co and Al should be visible. In our case, they remain undetected, which could show that either these species are not evaporated or the amount of Al is too low to be measured by OES. Due to the

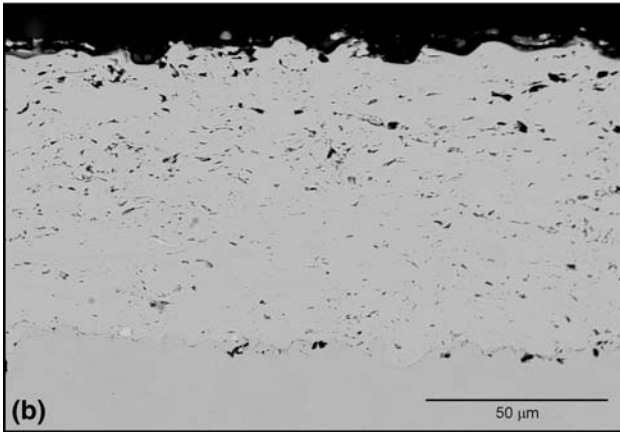
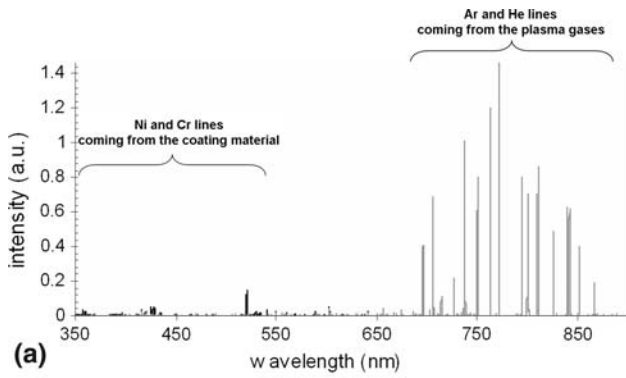


Fig. 12 (a) Emission lines of the plasma gas and metallic powder only partially in vapor phase using standard LPPS, and (b) splat-like microstructure of a MCrAlY coating produced with the same parameter without heat treatment

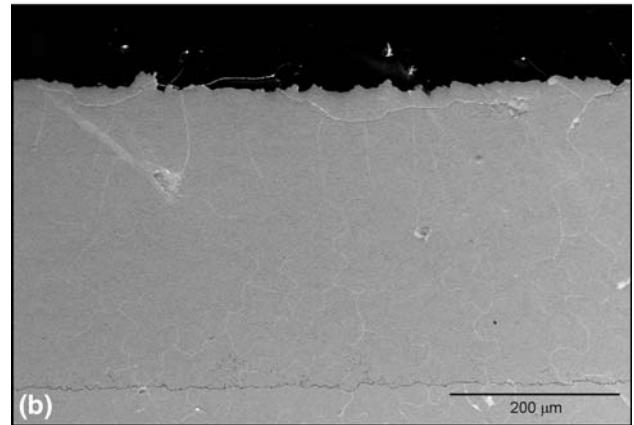
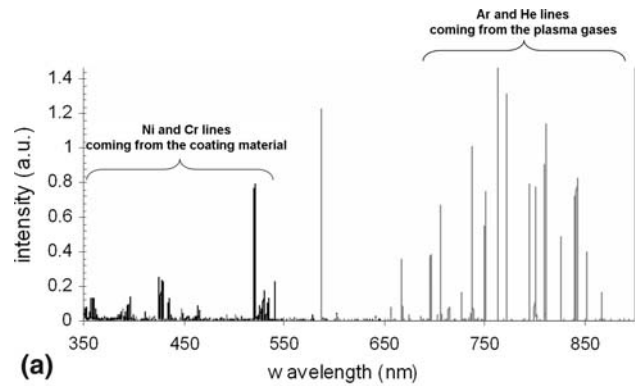


Fig. 13 (a) Emission lines of metallic powder in vapor phase using PS-PVD, and (b) dense oxide free MCrAlY coating produced with the same parameter without heat treatment

complexity of the spectral analysis of MCrAlY alloys in the vapor phase, these studies and the precise chemical composition of the coatings are still under investigation.

Despite the fact that with PS-PVD much denser and coatings with at least a very low oxide content can be achieved and that a high amount of the injected feed stock material is being vaporized, it is difficult to judge whether the deposited coating comes from the vapor phase or not. SEM investigations on such coatings still show splat-like microstructures even though the spectrum shows rich and intense Cr and Ni lines. Figure 15 shows a picture of the same MCrAlY coating (Amdry 365) deposited with PS-PVD using parameters giving intensive metal vapor emission lines without heat treatment. The splat-like structure of the coating is easily visible. Therefore, the measured vapor fraction of the coating material during deposition did not play an observable part in the coating build up.

4. Summary

The PS-PVD technology uses a reduced ambient pressure down to 1 mbar and an increased power level of a

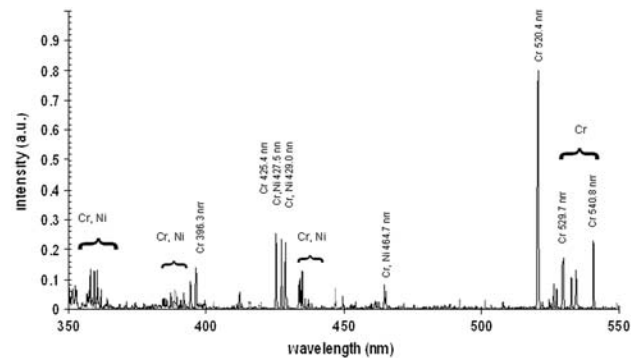


Fig. 14 Emission lines representing vaporized metal powder

modified plasma gun to achieve special plasma characteristics. With this technology, the injected powder material is not only molten into liquid droplets, but also evaporated. In the case of YSZ, coatings could be produced from the vapor phase which shows a unique columnar microstructure, so far only known from other vapor phase deposition processes like EB-PVD. The single columns consist of many fine needles with a high defect density and a high amount of internal porosity. With this

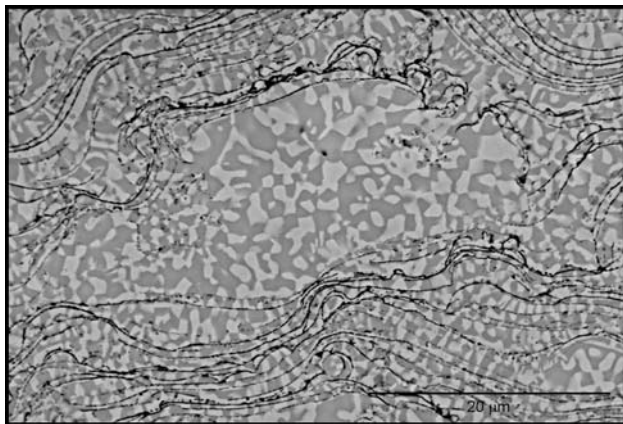
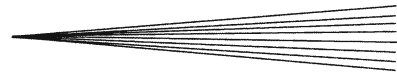


Fig. 15 SEM pictures of a MCrAlY coating without heat treatment, deposited using PS-PVD conditions and a parameter with intensive metal vapor emission lines

morphology, the coatings have some special characteristics which are advantageous for gas turbine applications. These PS-PVD produced coatings have proven to be high strain tolerance in FCTs and offer a low thermal conductivity. The erosion resistance achieved so far is still lower than EB-PVD coatings but has been significantly improved by an adequate process optimization. Real gas turbine substrates were coated to demonstrate the ability of PS-PVD for the deposition of such coatings onto components with complex shapes. Ongoing economical evaluations for columnar coatings on turbine components under production conditions show a clear saving potential when using the PS-PVD process. This technology offers a high potential to support the development of new coating solutions for improved TBC systems and their application under production relevant conditions with an increased economical benefit. The deposition of metallic layers out of the vapor phase is still under investigation and showed initial positive results.

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