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Suspension Plasma Spraying: Process Characteristics and Applications

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Suspension plasma spraying (SPS) offers the manufacture of unique microstructures which are not possible with conventional powdery feedstock. Due to the considerably smaller size of the droplets and also the further fragmentation of these in the plasma jet, the attainable microstructural features like splat and pore sizes can be downsized to the nanometer range. Our present understanding of the deposition process including injection, suspension plasma plume interaction, and deposition will be outlined. The drawn conclusions are based on analysis of the coating microstructures in combination with particle temperature and velocity measurements as well as enthalpy probe investigations. The last measurements with the water cooled stagnation probe gives valuable information on the interaction of the carrier fluid with the plasma plume. Meanwhile, different areas of application of SPS coatings are known. In this paper, the focus will be on coatings for energy systems. Thermal barrier coatings (TBCs) for modern gas turbines are one important application field. SPS coatings offer the manufacture of strain-tolerant, segmented TBCs with low thermal conductivity. In addition, highly reflective coatings, which reduce the thermal load of the parts from radiation, can be produced. Further applications of SPS coatings as cathode layers in solid oxide fuel cells (SOFC) and for photovoltaic (PV) applications will be presented.

Keywords photovoltaic, solid oxide fuel cells, suspension plasma spraying, thermal barrier coatings

1. Introduction

In the field of surface coating, the atmospheric plasma spraying (APS) is since several decades a well-established technology with many improvements especially with respect to plasma gun technology. Although a rather wide flexibility of coating morphologies can be obtained with this process, within the coatings typically the size of microstructural features is dictated by the used feedstock. This is for conventional plasma spraying a well flowable powder in the size range of about 10 to 100 μm. Also, the minimum thickness of the coatings is limited to about 10 μm, as at least several piled up splats should form the coating. Here, the use of liquid feedstock as solutions or suspensions yields a higher flexibility, even nanophase materials can be processed or synthesized within the process (Ref 1-3). Molten droplets with a diameter of a few hundred nanometres to a few micrometers can be generated. These fine droplets lead to much smaller splats compared to conventional APS splats. Furthermore, it is possible to generate completely new or at least improved coating structures. So it is not surprising that a wide range of research has been done on the suspension plasma spraying. Major fields of research are the generation of improved thermal barrier coatings (TBC) for gas turbines and engines, coatings for wear protection, components for fuel cells (SOFC), biocompatible coatings for implants, and silicon-free solar plants (Ref 4-7). Despite of the wide range of application and studies on the SPS process (Ref 8, 9), coating build-up is often only partly understood. This might lead to coatings with insufficient mechanical, optical, or physical properties. Also, the process efficiency and the reproducibility are affected.

Certainly, the used injection technology for the liquid plays an important role on the development of the process. In principle, two different ways exist: first, the direct injection of a stream of liquid is applied. Typically, this liquid will disintegrate during flight and will be further atomized in the plasma plume. The second possibility is the atomization of the liquid before injection. A good control of the atomization process is enabled by this method and therefore this method is used in our institute in most cases. However, a successful application necessitates sufficiently high droplet velocities. In addition to the proper injection technology, also the interaction between liquid and plasma plume might influence the deposition process. An investigation of this interaction is performed by the enthalpy probe. The enthalpy probe is a water

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jacketed gas sampling and stagnation probe for studying the enthalpy, temperature, and the velocity distribution in the hot and plasma gas flow. Furthermore, the measured gas can be sampled in order to analyze its composition by integration of a mass spectrometer. This technique was applied to the suspension plasma spray process to improve the understanding of the effects taking place in the plasma plume due to the injection of suspension fluids.

Enthalpy probes have been developed in the 1960s (Ref 10) and their applicability has been approved in many cases (Ref 11). Examples are the investigation of the impact of the ambient atmosphere on the characteristics of the plasma jet (Ref 12), the understanding of non-equilibrium situations in plasma jets (Ref 13), demixing effects and entrainment of surrounding cold gas with effect on plasma-particle interaction (Ref 14), nozzle design and optimization (Ref 15), oxidation control in different plasma gas compositions (Ref 16), and the characterization of new plasma gun concepts (Ref 17).

Regarding the suspension plasma spray process, preliminary results were achieved on the effect of argon carrier gas flow, ethanol, and water injection on the characteristics of an Ar/H_2-plasma with an F4 gun (Ref 18). It was found that in contrary to the carrier gas flow which hardly influences the plasma jet characteristics, the measured plasma gas temperatures and velocities were significantly affected by the injection of liquids. Thus, the injection of ethanol introduces an additional enthalpy source as its combustion heat is considerably above its evaporation enthalpy. Furthermore, the resulting temperature increase was found to depend on the depth of injection into the plasma jet. Regarding plasma velocity, the injection of ethanol led to a gain of momentum and thus to higher speeds. Again, the effect resulted to be dependent on the depth of injection.

Finally, the gun technology used is also important. In the present paper, only results from experiments made with TRIPLEX guns are presented. The reduced fluctuations in this type of guns seem to be advantageous for the suspension plasma process.

2. Experimental

A hydraulic circuit serves as feeding system for the suspensions. In most cases, a gas pressure of 0.2 MPa was used for the feeding, The injection was performed by a two-phase atomizer. This atomizer was designed to generate high velocities of the droplets at rather low liquid feeding rates. This is helpful for an efficient injection of the droplets into the plasma. The used pressures for atomization were about 0.1 MPa. The coatings were produced using a Triplex II APS gun from Sulzer Metco AG, Wohlen, Switzerland. For the deposition of thermal barrier coatings, rather high gun currents of 500 A and low stand-off distances below 80 mm were used, while more moderate conditions were employed for the deposition of the Graetzel cells, i.e. gun current 400 A and below, stand-off distance 130-140 mm.

Different types of substrate materials as sand-blasted austenitic steel (for basic investigations), bond coated nickel-based superalloys (for TBC systems), glass substrates (photovoltaic Graetzel cells), or electrolyte-coated anode substrates (SOFC) were used depending on the application.

Similarly different types of powders were used as partial yttria stabilized zirconia (5YSZ) from Tosoh Corporation, Tokyo, Japan, spray dried perovskite materials produced in-house and titania (anastase phase, Inframat, USA). The different powders were ball-milled by a grinding stock of zirconia balls to obtain particle sizes in the suspension below 1 μm. For the measurement of particle size in suspension, the DT1200 (Dispersion Technology, Bedford Hills, NY), an electroacoustic spectrometer was used. The suspension was prepared by dispersing the powder in ethanol. The mass content varied between 0.2 and 30 wt.%, typically 20 wt.% was used. The suspension was stabilized by the addition of 1.5 wt.% of an imine-based dispersant.

Speed and temperature monitoring of the particles were performed by the particle diagnostic system Accuraspray-g3, Tecnar, St. Bruno, Canada (Ref 19). For every measurement of the particle velocity and temperature, the measure time was at least 60 s.

Typically, the measurement of particle properties by standard single particle diagnostic systems as DPV2000 becomes increasingly difficult with decreasing particle size as the signal per individual particle is reduced. On the other hand, techniques measuring ensemble properties as the Accuraspray can evaluate properties for particle finer than 10 μm. Nevertheless, it should be noted that an assumption used here for the comparison of conventional and suspension plasma spraying is that the measurements are hardly effected by the different particles sizes and the liquid addition.

The microstructure was characterized by light microscopy and scanning electron microscopy (Ultra 55, Carl Zeiss NTS AG, Germany). Thereby, cross-sections, fracture surface, and the coating surface were examined.

The SPS enthalpy probe measurements were performed with a Triplex I plasma gun on an A3000 atmospheric plasma spray facility supplied by Sulzer Metco AG, Wohlen, Switzerland. For these measurements, only the carrier liquid without particles was injected instead of suspension. A hydraulic circuit as mentioned above served as feeding system. The injection was made in a 90° angle relative to the torch axis. The plasma gas composition was 40 slpm Ar and 10 slpm He, a rather typical composition for the Triplex torch, the current was 250 A (power 17.3 kW). The enthalpy probe system (ENP-04-CS, Tekna Plasma Systems Inc., Sherbrooke, QC, Canada) was used to determine the local enthalpy, temperature, velocity, and gas composition of the plasma. It is a combination stagnation probe and flowing calorimeter. The probe itself consists of a double wall tubing, which is cooled by a high-pressure water circuit. The outer diameter of the appropriate probe tube was 4.76 mm and the inner diameter 1.27 mm. The cranked tip was radially immersed into the plasma plume. All measurements were performed close to isokinetic conditions. This means that the gas velocity at the probe entrance is kept similar to the free stream velocity by adjusting the sampling rate. The shortest measurement distance to the nozzle was 50 mm to avoid thermal overload of the probe tip by excessive local heat flux.

To analyze its composition, the sampled gas was directly piped to a quadrupole mass spectrometer (Prisma QMS 200, Balzers AG, Balzers, Liechtenstein), which was calibrated by some known gas mixtures. Based on the actual plasma gas composition, the enthalpy can be calculated from the temperature increase of the cooling water between feed and return of the probe (Ref 20, 21). Moreover, the plasma velocity was determined from the measured stagnation pressure by the Bernoulli's equation.

3. Results and Discussion

3.1 Basic Studies

Typical values of particle velocities and temperatures of droplets for atmospherically sprayed YSZ suspensions are shown in Fig. 1.

The highest particle temperatures and velocities can be achieved at rather low spraying distances. Velocities of above 400 m/s are observed which can hardly be achieved with the used Triplex II gun for conventional powder feeding if in parallel also sufficiently high particle temperatures (above 2700 °C in case of YSZ) should be achieved. Also, rather high particle temperatures above 2500 °C are observed for the short spraying distance and the high current. However, the temperatures for conventional spraying are still higher (above 3000 °C).

It is interesting to note that for conventional plasma spraying with the Triplex II torch and the given operational conditions, typically a maximum in velocity and temperature of the particles is obtained at spraying distances around 90 and 100 mm. In SPS this distance is much shorter and the need to use low spraying distances for high deposition rates has also been observed by other groups (Ref 22). In conventional spraying, the maximum can be correlated with the decrease of velocity and temperature in the plasma plume mainly due to entrainment of air.

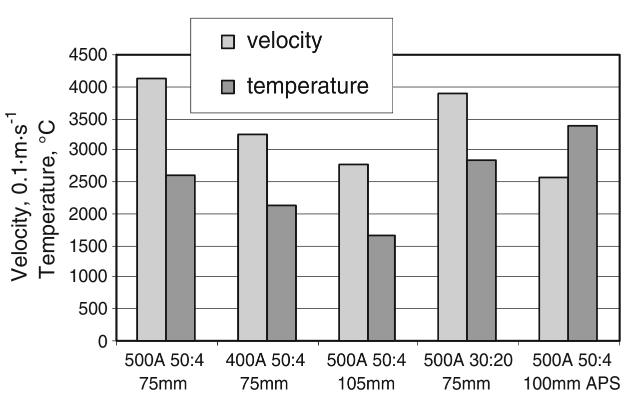

Fig. 1 Velocities and temperatures of droplets in the SPS process for different spray conditions (plasma current, Ar/He in slpm and stand-off distance). For comparison also data for a conventional YSZ powder are given (Ref 19)

An explanation of the reduced location of the maximum in SPS has to deal separately with temperature and velocity. Looking at the velocity at spraying distances of 105 mm for SPS and 100 mm for APS very similar values are found: both types of particles have reached the gas velocity. Going to smaller spraying distances, the gas velocity increases, the small SPS particles can reach these high velocities due to their low inertia, hence giving an increased velocity at lower spraying distances. The larger APS particles cannot be accelerated during the reduced flight time to values close to the gas velocity.

For the temperatures, the findings are different as here the value for the SPS is at the 100/105 mm spraying distance much lower (1600 °C compared to 3400 °C). Cooling sets in when the heat flux from the plasma is equal to the radiative losses, i.e.

$$\alpha(T - T_p) = \varepsilon\varepsilon_r T_p^4 \qquad \text{(Eq 1)}$$

with α the heat transfer coefficient (about 2 * 10^4 $W/m^2/K$ (Ref 10), T plasma temperature and T_p particle temperature, ε Stefan Boltzmann constant = 5.67 * 10^{-8} $W/m^2/K^4$, and ε_r emissivity ~0.2 (Ref 23, 24). Looking at the order of magnitudes of heat transfer and emissivity coefficients, even at low temperature differences the convective heating is still dominating over the radiation losses. It is also obvious that radii do not play a role for this estimation. Hence, the plasma temperature profile seen by the droplets has to be different in SPS, i.e. lower, to explain the different values. The reason for this might be an insufficient penetration into the plasma or diffusion out of the center by thermophoretic forces (Ref 25): the last ones are becoming considerable for increasing surface to volume ratios. For example, in a 10,000 K plasma, the ratio of the vertical force (resulting from gravitation and in opposite direction from thermophoresis) and the particle mass increases for more than two magnitudes for a 1 µm particle compared to a 20 µm one. Thus, in SPS processes, smaller particles are accelerated radially away from the hot plasma core, leading to reduced maximum particle temperatures.

A cooling of the plasma plume by the liquid and air injection is not likely as the results presented below indicate an even higher plasma temperature for spraying distances above 50 mm.

Although the temperature seems to be reduced, an optimization of the process conditions (higher He gas flow, Fig. 1) can further improve the temperature, leading to both high temperatures and velocities of the SPS particles.

These particle properties indicate that the heating and acceleration of the droplets in the plasma plume is efficient which also proves the efficiency of the injection system used.

3.2 Enthalpy Probe Measurements

Figure 2 shows the measured plasma gas temperatures for three cases: (i) APS: without any injection; ii) SPS air + eth: two-phase injection of ethanol and air; (iii) SPS air only: injection of air without ethanol.

The injection of air cools down the plasma plume. Furthermore, the radial temperature distribution is shifted downwards. At the axial distance of 50 mm, the maximum is moved down for approximately 5 mm. However, this cooling effect appears quite moderate, which proves the feasibility of the newly designed two-phase atomizer. If ethanol is added, the temperatures are enhanced considerably. The maximum temperature at nozzle distance of 50 mm rises from 2458 K (air injection only) to 3487 K with ethanol. This is due to its combustion enthalpy of 29,800 kJ/kg which is much more than the vaporization enthalpy of 840 kJ/kg (Ref 25). In the case of ethanol injection, the plasma gas shows a distinct CO_2 content while the oxygen fraction is reduced by the combustion. In axial direction, the maximum CO_2 content of 2.65 vol.% is reached at a distance of 70 mm, indicating that the combustion of the ethanol is completed here. At the same point, the oxygen content is lowered by the combustion from 19.5 vol.% (air injection only) to 13.8 vol.% with ethanol.

Figure 3 shows the measured velocity distributions in the plasma gas for the three cases mentioned above. In principle, the same characteristics as the temperatures are observed. Due to the air injection, the velocities measured at a nozzle distance of 50 mm are lowered from 379 m/s (no injection) to 336 m/s (air injection only). The combustion of ethanol leads again to an increase to 402 m/s. These velocities are in the same range as the particle velocities (Fig. 1); however, a direct comparison is not possible as a different gun was used.

4. Applications

4.1 Thermal Barrier Coatings (TBCs)

Since several decades thermal barrier coatings have been used to protect components in the hot gas stream of gas turbines. Different types of structures have been established as micro-cracked and segmented coatings produced by conventional atmospheric plasma spraying (Ref 26). Segmented TBCs offer a good thermal shock resistance (Ref 27); however, the thermal conductivity of the coatings is significantly higher than the one of conventional coatings (typically more than 50% higher).

With suspension plasma spraying it is now possible to obtain segmented coatings with high segmentation crack densities and low thermal conductivity. The reason is the different relaxation processes during spraying. While splat cooling in conventional plasma spraying leads to high amount of micro-cracks, a reduced amount of these cracks is observed in SPS coatings. This is obvious from a comparison of the two micrographs shown in Fig. 4. It can be

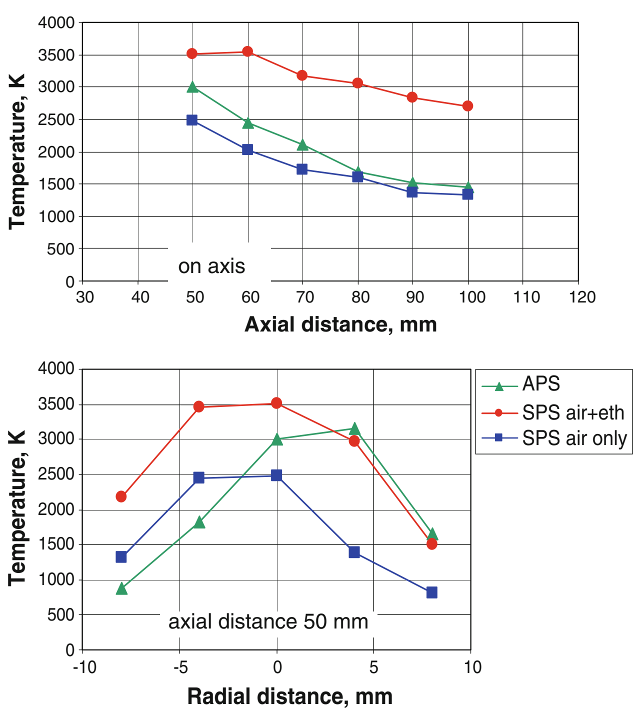

Fig. 2 Axial and radial distributions of the measured plasma gas temperatures for three cases (APS: without any injection, SPS air + eth: two-phase injection of ethanol and air, SPS air only: injection of air without ethanol)

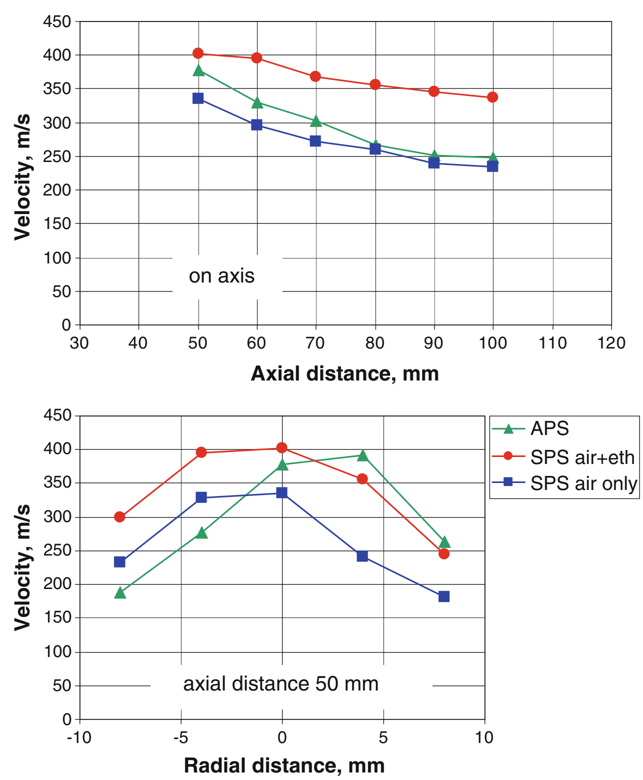

Fig. 3 Axial and radial distributions of the measured plasma gas velocities for three cases (APS: without any injection, SPS air + eth: two-phase injection of ethanol and air, SPS air only: injection of air without ethanol)

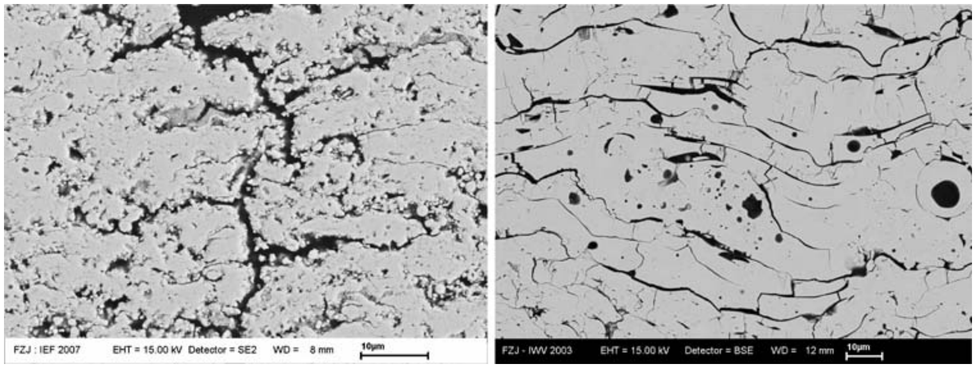

Fig. 4 Micrographs of a suspension plasma sprayed thermal barrier coating (left) showing a segmentation crack and a micro-cracked conventional APS TBC (right)

explained by the energy release rate during cooling which tends to decrease for thinner splats, giving not sufficient energy for crack propagation. Hence, the SPS coating contains a lower amount of micro-cracks. These cracks can relax tensile stress levels build up during splat cooling. On the other hand, the tensile stress is essential for segmentation crack formation. As a result, SPS coatings form more easily segmentation cracks and can do this even for more porous structures.

The segmented SPS coatings were tested in furnace and cyclic burner test rigs and showed a very good performance (Ref 28). More details will be published elsewhere.

The reflectivity of the SPS coatings were found to be higher as the one of conventional coatings. The transmittance was reduced from more than 10% to less than 3% for the SPS coatings (Ref 29) because more scattering centers are introduced. In a gas turbine environment, this effect would reduce effectively the direct heating of the components by radiation from the hot combustion gases and the other hot components.

4.2 Solid Oxide Fuel Cells (SOFC)

Solid oxide fuel cells are very efficient energy conversion systems even for rather small units in the kW range. A major obstacle for a wide spread utilization is besides long-term stability the costs of the systems, so different manufacturing routes have been tested, also thermal spray routes (see, e.g., Ref 30). Especially for the cathode deposition, SPS seems to be a suitable process as coatings with high porosity levels in combination with fine pore size can be produced (Ref 31). Such microstructures are advantageous for a good electrochemical performance. Figure 5 shows such a coating with fine pores in the sub-micrometer range and a high porosity level of about 30%.

On the other hand, it turned out that also other deposition techniques as screen printing can offer advantages with respect to cost-efficiency and performance (Ref 32).

4.3 Photovoltaic Cells

Recently, SPS has been used as a deposition technique for TiO_2 layers in Graetzel cells (Ref 33).

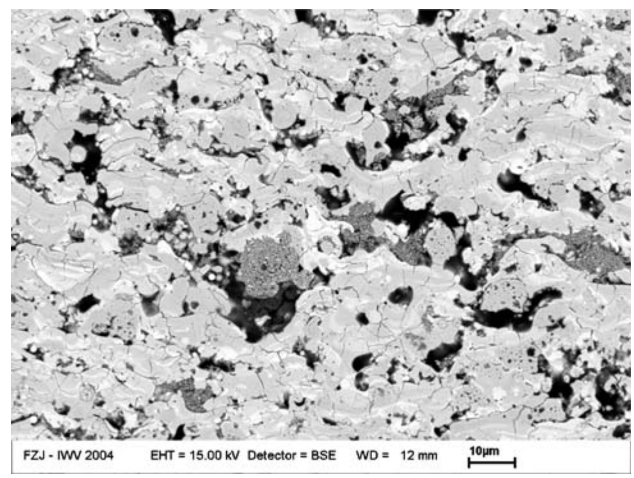

Fig. 5 Microstructure of a LSM cathode layer produced by SPS

Also, in this case, the coating should have a high surface area and a very fine grain structure which can be obtained by SPS. The SPS process also allowed the manufacture of coatings with a very high amount of anatase phase (about 90%, Ref 34). Up to now, the performance of our cells is lower than those prepared by sintering techniques. However, we could improve our cells by both the contacting and the geometry optimization giving values above 1 mW/cm^2. Further improvements are expected by process optimization and better dyes.

5. Conclusion

Measurements of velocities and temperatures of the suspension droplets indicate a very high velocity, however a reduced temperature. Also, the maximum in temperature and velocity occurred at significantly lower spraying distance compared to the conventional spray process. The enthalpy probe measurements showed that the plasma gas characteristics were considerably affected by the carrier fluids of suspensions. The energy absorption for heating

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and vaporization was compensated by the ethanol combustion enthalpy. By contrast, plasma temperatures and velocities were considerably enhanced compared to the case of no injection. The use of a two-phase atomizer may be advantageous because oxygen for combustion was provided immediately after injection. Thus, combustion was not limited to the downstream parts of the plume until oxygen was entrained from the surrounding atmosphere. The two-phase atomizer should make the SPS process considerably more effective.

The use of suspensions in thermal spray processes leads to coatings with new microstructures and often improved properties compared to conventional sprayed coatings. As a result, meanwhile, a number of possible applications for suspension plasma spraying exist especially in the field of energy conversion systems (e.g., TBCs, SOFCs, PV).

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